DEPARTMENT OF THE AIR FORCE
AIR FORCE CIVIL ENGINEER CENTER

July 24, 2015

MEMORANDUM FOR: U.S. Environmental Protection Agency – Region 2
Attn: Robert Morse
Federal Facilities Section
290 Broadway, 18 Floor
New York, NY 10007-1866

New York State Department of Environmental Conservation
Attn: Ms. Heather Bishop
Division of Environmental Remediation
625 Broadway 11th Floor
Albany, NY 12233-7015

Mr. Mark Tibbe
NYS Department of Environmental Conservation
207 Genesee Street
Utica, New York 13501-2885

FROM: AFCEC/CIBE – Plattsburgh
8 Colorado Street, Suite 121
Plattsburgh NY, 12903

SUBJECT: Final 2015 Update Uniform Federal Policy Quality Assurance Project Plan
Former Griffiss Air Force Base (AFB) Rome, New York
Contract Number FA8903-10-D-8595
Delivery Order 0014
May 2015

Accompanying this letter please find the "Final 2015 Update Uniform Federal Policy Quality Assurance Project Plan", for your review and comment. The Draft 2015 Update Uniform Federal Policy Quality Assurance Project Plan was submitted on May 12, 2015.

We would appreciate review comments by August 24, 2015 so that project schedules and performance milestones can be maintained in accordance with this PBR Contract.

Should you have any questions or concerns please contact me at 518-563-2871.

David S. Farnsworth
Program Manager/BRAC Environment Coordinator
BRAC Program Execution Branch

FINAL

2015 UPDATE
UNIFORM FEDERAL POLICY
QUALITY ASSURANCE PROJECT PLAN

FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Prepared for:

Air Force Civil Engineer Center
Building 171
2261 Hughes Avenue, Suite 155,
Joint Base San Antonio Lackland, TX

Prepared by:

FPM

FPM Remediations, Inc.
584 Phoenix Drive
Rome, NY 13441

In association with:

CAPE

10901 Lowell Ave., Ste. 271
Overland Park, KS 66210

AECOM

40 British American Boulevard,
Latham, NY 12110

Contract Number FA8903-10-D-8595
Delivery Order 0014

July 2015

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Acronyms and Abbreviations

%D	Percent Difference
AECOM	AECOM Technical Services
AFB	Air Force Base
AFCEC	Air Force Civil Engineer Center
AFCEE	Air Force Center for Engineering and the Environment
AOC	Area of Concern
AOI	Area of Interest
BRAC	Base Realignment and Closure
BOD	Biological Oxygen Demand
°C	degrees Celsius
CA	Corrective Action
CAPE	CAPE, Inc.
CAS	Chemical Abstracts Service
CCC	Calibration Check Compounds
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CV	Calibration Verification
CFR	Code of Federal Regulations
CLLE	Continuous Liquid/Liquid Extraction
COC	Chain of Custody
COD	Chemical Oxygen Demand
DL	Detection Limit
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
EICP	Extracted ion current profile
ERPIMS	Environmental Restoration Program Information Management System
EXC	Capital Investment Execution Division
FDA	Fire Demonstration Area
FFA	Federal Facilities Agreement
FPM	FPM Remediations, Inc.
FPTA	Fire Protection Training Area
GC	Gas Chromatography
GC/MS	Gas Chromatography and Mass Spectrometer
GLDC	Griffiss Local Development Corporation
HCl	Hydrochloric Acid
H&S	Health and Safety

ICV	Initial Calibration verification
ID	Identification
IRP	Installation Restoration Program
IS	Internal Standard
kg	kilogram(s)
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LTM	Long Term Monitoring
LUC/IC	Land use Control/Institutional Control
mg	milligram(s)
mL	milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
N/A	Not Available
No.	Number
NPL	National Priorities List
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
O&M	Operation and Maintenance
oz	ounce
PAH	Polynuclear Aromatic Hydrocarbon
PCBs	Polychlorinated Biphenyls
PID	photoionization detector
PGM	Program Manager
pH	Measure of the acidity or basicity of a solution (pH = -log[hydrogen ion concentration])
PM	Project Manager
PMP	Project Management Plan
POC	point of contact
POP	Period of Performance
ppm	parts per million
PQL	Practical Quantitation Limit
PQO	Practical Quality Objective
PVC	Polyvinyl Chloride
QA	Quality Assurance

QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
r	Correlation Coefficient
r^2	Coefficient of Determination
RACR	Remedial Action Completion Report
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
STD	Standard
SVE	Soil Vapor Extraction
SVI	Soil Vapor Intrusion
SVOC	Semi-Volatile Organic Compound
TA	Test America Laboratories, Inc.
TAL	Target Analyte List
TBD	To Be Determined
TDS	Total Dissolved Solids
TIC	Tentatively Identified Compound
TKN	Total Kjehldahl nitrogen
TOC	Total Organic Carbon
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
UST	underground storage tank
USEPA	United States Environmental Protection Agency
VES	Vapor Extraction System
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
µg/L	microgram per liter

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INTRODUCTION

FPM Remediations, Inc. (FPM), in association with CAPE Inc. (CAPE) and AECOM Technical Services (AECOM), under contract with the Air Force Civil Engineer Center (AFCEC), is performing long term monitoring (LTM), site remediation, and site investigations at the former Griffiss Air Force Base (AFB), Rome, New York (Figure 1-1).

The former Griffiss AFB covered approximately 3,552 contiguous acres in the lowlands of the Mohawk River Valley in Rome, Oneida County, New York. Topography within the valley is relatively flat, with elevations on the former Griffiss AFB ranging 435-595 feet above mean sea level. Three Mile Creek, Six Mile Creek (both of which drain into the New York State Barge Canal, located to the south of the base), and several state-designated wetlands are located on the former Griffiss AFB, which is bordered by the Mohawk River on the west. Due to its high average precipitation and predominantly silty sands, the former Griffiss AFB is considered a groundwater recharge zone.

The scope of work to be completed for this project is summarized in Table 1-1.

TABLE 1-1
GRIFFISS PERFORMANCE BASES REMEDIATION SCOPE OF WORK

Group	Sites	Work Element	Monitoring Matrix	Site Objective	Site Update Year 2014
CERCLA Sites – Landfill Areas of Concern (AOCs)	LF001, LF002, LF003, LF007, and LF009	LTM	Groundwater and Surface Water	Optimization	Optimization
CERCLA Sites - SD052 SVI System Sites	Buildings 774, 776, 785, and 786	SVI System Operation and Monitoring	Indoor/Outdoor Air and Sub-Slab Vapor	Optimization	Optimization
NYSDEC Petroleum Spill Sites – Site Closures	SS064	Groundwater/ Soil Remediation* and LTM	Groundwater Groundwater and Surface Water at SS064	Site Closure	Site Closure
NYSDEC Petroleum Spill Sites – Site Optimization	SS054 and SS067	Groundwater/ Soil Remediation and Long Term Monitoring	Groundwater	Optimization	Optimization

Group	Sites	Work Element	Monitoring Matrix	Site Objective	Site Update Year 2014
NYSDEC Petroleum Spill Sites – Site Optimization	Building 785 Pipeline	Groundwater/ Soil Remediation and Sampling	Groundwater and soil	remediation and performance monitoring	Unknown site to undergo remediation and one performance monitoring event
CERCLA Sites – SVI Land use Control/Institutional Control (LUC/IC) Sites	ST006	Sampling	Indoor/Outdoor Air, Sub-Slab Vapor, and Soil Vapor	Site Closure	Site part of PBR initially for LUC/ICs inspections only. Objective changed to Site Closure in 2012 Contract Modification

Note:
* - Groundwater and Soil Remediation will not occur at SS070.
NYSDEC = New York State Department of Environmental Conservation
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
SVI = Soil Vapor Intrusion

This Uniform Federal Policy Quality Assurance Project Plan (UFP/QAPP) has been prepared in conjunction with the tasks described in the Former Griffiss AFB 2014 Updated Project Management Plan (PMP) (CAPE, September 2014) and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Sites and Petroleum Spill Sites Optimization Plans.

Quality Assurance Project Plan (QAPP) Worksheet #1 – Title and Approval Page

UFP-QAPP
Document Title

Air Force Civil Engineering Center
Lead Organization

Daniel Baldyga, FPM Remediations, Inc.
Preparer's Name and Organizational Affiliation

584 Phoenix Drive, Rome, NY, 13441, (315)336-7721, d.baldyga@fpm-remediations.com
Preparer's Address, Telephone Number, and E-mail Address

03/10/2015
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: Phil Dula, Project Manager (PM)
Printed Name/Title

Signature/Date

Investigative Organization's Project Quality Assurance (QA) Officer:
Henry Vaca, Chem. Quality System Manager
Printed Name/Title

Signature/Date

Lead Organization's Project Manager: David Farnsworth, AFCEC PM
Printed Name/Title

Signature/Date

Laboratory QA Manager: Elaine Walker, Test America (TA)
Printed Name/Title

Signature/Date

Other Approval Authority: Mike Healy/General Operations Manager
Printed Name/Title

Signature/Date

QAPP Worksheet #2 – QAPP Identifying Information

Site Number/Code: None

Operable Unit: None

Contractor Name: CAPE/FPM/AECOM

Contractor Number: FA8903-10-D-8595

Contract Title: Performance Based Remediation

Work Assignment Number: Task Order

1. Identify guidance used to prepare QAPPs:
   UFP QAPP and Air Force Center for Engineering and the Environment (AFCEE) QAPP Version 4.0.02, May 2006.

2. Identify regulatory program:
   CERCLA, Resource Conservation and Recovery Act, United States Environmental Protection Agency (USEPA) Region II, and New York State Department of Environmental Conservation (NYSDEC)

3. Identify approval entity:
   AFCEC, USEPA, NYSDEC

4. This is a project-specific QAPP. This document was prepared in conjunction with the Former Griffiss AFB 2014 Updated PMP (CAPE, September 2014) and CERCLA Sites and Petroleum Spill Sites Optimization Plans.

5. List dates of scoping sessions that were held: March 30, 2011 and March 31, 2011.

6. List dates and titles of QAPP documents written for previous site work, if applicable:
   Basewide AFCEE QAPP for the former Griffiss AFB, October 2006.

7. List organizational partners (stakeholders) and connection with lead organization: USEPA, regulator; NYSDEC, regulator; New York State Department of Health (NYSDOH), regulator; AFCEC, representative party; CAPE, prime contractor; FPM, sub-contractor; AECOM, sub-contractor; and Property Owners/Occupants (including the Griffiss Local Development Corporation (GLDC), Mohawk Valley EDGE, and Oneida County Department of Aviation).

8. List data users:
   AFCEC, USEPA, NYSDEC, and NYSDOH

   If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion: Not applicable.

QAPP Worksheet #3 – Distribution List

QAPP Recipients	Title	Organization / Address	Telephone Number	E-mail Address	Document Control Number
David Farnsworth	Griffiss BRAC Manager	Air Force Civil Engineer Center	518-563-2871	david.farnsworth@us.af.mil	
Robert Morse	Remedial PM	United States Environmental Protection Agency	212-637-4331	Morse.Bob@epamail.epa.gov	
Heather Bishop	Environmental Engineer	New York State Department of the Environmental Conservation	518-402-9692	hlbishop@gw.dec.state.ny.us	
Mark Tibbe	NYSDEC Petroleum Spills	New York State Department of the Environmental Conservation	315-793-2554	mctibbe@gw.dec.state.ny.us	
Kristin Kulow	NYSDOH	New York State Department of Health	607-432-3911	kxk07@health.state.ny.us	
Mike Healy	General Manger of Operations	CAPE Inc.	847-548-5994	mhealy@cape-inc.com	
Phil Dula	PM	CAPE Inc.	913-327-8300	pdula@cape-inc.com	
Gaby Atik	FPM Task Manager	FPM Remediations, Inc.	315-336-7721	g.atik@fpm-remediations.com	
Mike Niederreither	AECOM Task Manager	AECOM	717-790-3404	mike.niederreither@aecom.com	
Elaine Walker	Test America PM	Test America (Denver Laboratory)	303-736-0156	Elaine.walker@testamericainc.com	

QAPP Worksheet #4 – Project Personnel Sign-Off Sheet

Organization: AFCEC

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
David Farnsworth	AFCEC/CIBE Plattsburgh	315-356-0810		
Cathy Jerrard	AFCEC/ CIBE Griffiss	315-356-0810		

Organization: CAPE

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Kurt Gates	Program Manager (PGM)	210-377-2008		
Mike Healy	General Manager of Operations	847-548-5994		
Phil Dula	Project Manager	913-327-8300		
Merle Miller	Senior Engineer	210-377-2008		
Glen Mayekawa	Health and Safety (H&S) Manager	714-599-9071		
Henry Vaca	Quality System Manager	770-908-7200		

Organization: FPM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Gaby Atik	FPM Task Manager	315-336-7721		
Daniel Baldyga	FPM Technical Lead	315-336-7721		
Connie van Hoesel	Chemical Quality Control (QC) Coordinator	315-336-7721		

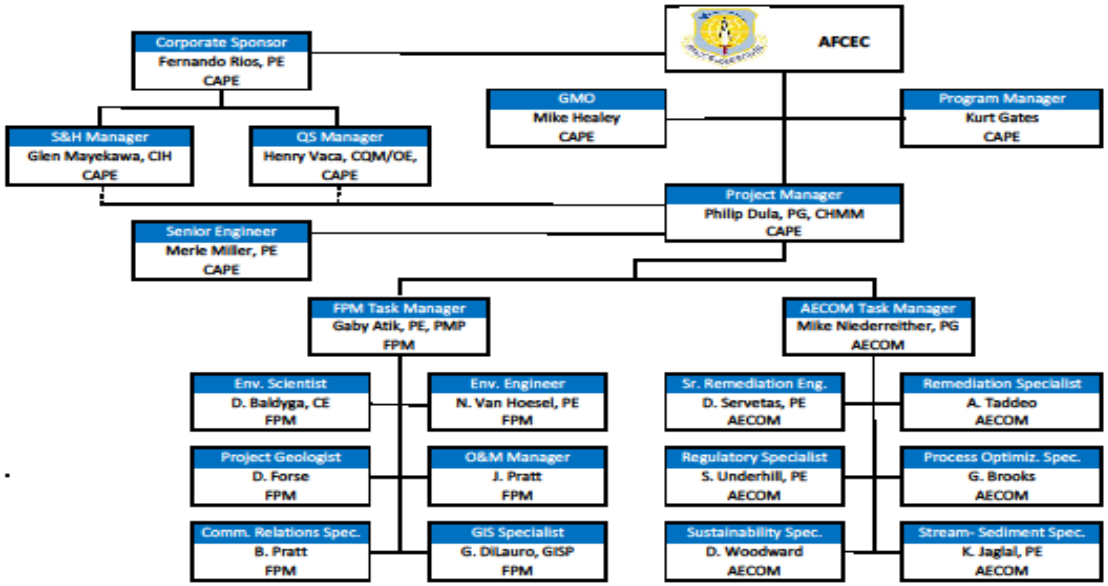
Organization: AECOM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Mike Niederreither	AECOM Task Manager	717-790-3404		
John Santacroce	AECOM Technical Lead	518-951-2265		

Organization: Laboratory (Test America)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Elaine Walker	TA PM	303-736-0156		

QAPP Worksheet #5 – Project Organizational Chart



QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, pathways, etc.)
Point of Contact (POC) with USEPA, NYSDEC, and NYSDOH	AFCEC/ CIBE Plattsburgh	David Farnsworth	518-563-2871	David Farnsworth is the Griffiss BRAC Base Environmental Coordinator (BEC)
	AFCEC/ CIBE Griffiss	Cathy Jerrard	315-356-0810	Ms. Jerrard is an Environmental Engineer for Griffiss Support and an alternate point of contact to David Farnsworth.
Overall Project Management	AFCEC/ CIBE Griffiss	David Farnsworth	518-563-2871	David Farnsworth is the Griffiss BEC
Manage Program	CAPE PGM	Kurt Gates	210-377-2008	Is the single POC with authority for the program organization.
Manage entire project	CAPE PM	Phil Dula	913-327-8300	Is the primary interface with AFCEC and ensures performance objectives are met.
Manage project team	General Manager of Operations	Mike Healy	847-548-5994	Develops and maintains alliances with Team subcontractors.
Manage FPM Tasks	FPM Task Manager	Gaby Atik	315-336-7721	Communicates with CAPE PM and provides monthly reports and schedule updates to CAPE PM
Manage AECOM Tasks	AECOM Task Manager	Mike Niederreither	717-790-3404	Communicates with CAPE PM and provides monthly reports and schedule updates to CAPE PM
QAPP Changes in the Field	FPM Technical Lead	Daniel Baldyga	315-336-7721	Supervises field sampling and Operation and Maintenance (O&M) activities.
Daily Field Progress Reports	FPM Technical Lead	Daniel Baldyga	315-336-7721	Supervises field sampling and O&M activities. Authors status and completion reports.
Sampling, Drilling, and Monitoring Well Installation	AECOM Technical Lead	John Santacroce	518-951-2265	Responsible for all drilling, sampling, and monitoring well installation activities to assure goals of the field investigations are attained.

QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Reporting Lab Data Quality Issues	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	Will determine corrective actions (CAs) for lab data quality issues
Field and Analytical CAs	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	Will determine CAs for field and analytical issues
Release of Analytical Data	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	No analytical data can be released until validation is completed and has approved the release.
QAPP Amendments	AFCEC/CIBE Griffiss	David Farnsworth	518-563-2871	Any major changes to the QAPP must be approved by David Farnsworth before the changes can be implemented.

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
David Farnsworth	Griffiss BEC	AFCEC/CIBE Plattsburgh	Support role as on site Air Force representative.	B.S. Civil Engineering, 32 years environmental experience with the federal government.
Cathy Jerrard	Environmental Engineer	AFCEC/CIBE Griffiss	Support role as on site Air Force representative.	B.S. Mechanical Engineering, 16 years of environmental experience.
Kurt Gates	PGM	CAPE	PGM	B.S., Safety Science Engineering, over 20 years of experience as PGM.
Phil Dula	PM	CAPE	Manages project – coordinates between lead agency and subcontractor.	M.B.A., M.S. Geology, B.A. Biology, 28 years environmental experience.
Mike Healy	General Manager of Operations	CAPE	Develops and maintains alliances with Team subcontractors.'	M.S. and B.S. Geology, 23 years environmental experience.
Merle Miller	Project Engineer	CAPE	Ensures all environmental, civil, and process engineering support goals are achieved.	B.S. Civil Engineering, 13 years of civil engineering experience.
Gaby Atik	FPM Task Manager	FPM	Communicates with CAPE PM and directs site work to ensure exact compliance with approved PWS, work plan, SSHP, CQC Plan, and applicable regulations.	M.S. Environmental Management, 20+ years environmental experience.
Mike Niederreither	AECOM Task Manager	AECOM	Communicates with CAPE PM and directs site work to ensure exact compliance with approved PWS, work plan, SSHP, CQC Plan, and applicable regulations.	M.S. Hydrology, B.S. Geology, 20+ years of environmental experience.
Daniel Baldyga	FPM Technical Lead	FPM	Supervises field sampling and coordinates all field activities.	B.S. Biology, 10+ years of environmental experience.
John Santacroce	AECOM Technical Lead	AECOM	Supervises field sampling and coordinates all	B.S, 10+ years of environmental

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
			field activities.	experience.
Glen Mayekawa	H&S Officer	CAPE	Oversees H&S for field activities.	M.S. and B.S. Health Science, 30+ years of comprehensive industrial hygiene experience and health and safety management.
Henry Vaca	Quality System Manager	CAPE	Quality System Manager	M.S. Quality Assurance, B.S. Mechanical Engineering, 25+ years of Quality Management Experience
Connie van Hoesel	Team Chemist/Data Specialist	FPM	Manages analytical laboratory (soil and groundwater samples).	M.S. Environmental Engineering, B.A. Chemistry, 11 years experience
Connie van Hoesel	Chemical Quality Control Coordinator	FPM	Determines need for CA for field and analytical issues. Performs data verification.	M.S. Environmental Engineering, B.A. Chemistry, 11 years of experience.

QAPP Worksheet #8 – Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training[1]	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[2]
Field chemistry and sampling	H&S Training per 29 Code of Federal Regulations (CFR) 1910.120 Confined Space Entry Training – 8-hour per 29 CFR 1910.146 Tailgate meeting to discuss sampling plan and procedures	Various	Various Start of fieldwork	All	FPM Technical Lead, FPM	Onsite office Safety File, office electronic backup

1 All sampling personnel will be trained using sampling techniques described in the SOP (Appendix A).

2 All field personnel (including drilling sub-contractors) certifications will be retained electronic by the CAPE team for review.

QAPP Worksheet #9 – Project Scoping Session Participants Sheet

Project Name: Former Griffiss AFB PBR **Projected Date(s) of Sampling:** January 1, 2011 through December 31, 2015 **Project Manager:** Phil Dula, CAPE	**Site Name:** Former Griffiss AFB Petroleum Spill sites and CERCLA AOCs **Site Location:** Former Griffiss AFB, Rome, New York

Date of Session: March 30 and 31, 2011

Scoping Session Purpose: Kickoff meetings with Regulatory agencies to discuss site remediation/monitoring approaches and site objectives.

March 30, 2011

Name	Title	Affiliation	Phone #	E-mail Address
Michael McDermott	Former AFCEC PM			
Cathy Jerrard	AFCEC-Griffiss	AFCEC	315-356-0810	catherine.jerrard@us.af.mil
Scott Reichinger	NYSDEC Petroleum Spills	NYSDEC	315-793-2554	screichi@gw.dec.state.ny.us
Mark Tibbe	NYSDEC Petroleum Spills	NYSDEC	315-793-2747	mctibbe@gw.dec.state.ny.us
Phil Dula	PM	CAPE	913-327-8300	pdula@cape-inc.com
Gaby Atik	FPM Task Manager	FPM	315-336-7721	g.atik@fpm-remediations.com
Daniel Baldyga	FPM Technical Lead	FPM	315-336-7721	d.baldyga@fpm-remediations.com
John Santacroce	AECOM Technical Lead	AECOM	518-951-2265	john.santacroce@aecom.com

March 31, 2011

Name	Title	Affiliation	Phone #	E-mail Address
Doug Pocze	Remedial PM	USEPA	212-637-4432	Pocze.Doug@epamail.epa.gov
Heather Bishop	Environmental Engineer	NYSDEC	518-402-9692	hlbishop@gw.dec.state.ny.us
John Swartout	NYSDEC	NYSDEC	518-402-9620	jbswarto@gw.dec.state.ny.us
Jim Harrington	NYSDEC	NYSDEC	518-402-9624	jbharrin@gw.dec.state.ny.us
Phil Dula	PM	CAPE	913-327-8300	pdula@cape-inc.com
Gaby Atik	FPM Task Manager	FPM	315-336-7721	g.atik@fpm-remediations.com
Daniel Baldyga	FPM Technical Lead	FPM	315-336-7721	d.baldyga@fpm-remediations.com
Daniel Servetas	Former AECOM Technical Lead	AECOM	518-951-2378	daniel.servetas@aecom.com

Consensus Decisions:

Kick-off Meetings

The former Griffiss AFB PBR kickoff meetings were held to discuss all proposed site activities and site objectives. The discussions were held for 2 days to obtain feedback from the regulatory agencies. The March 30, 2011, meeting addressed planned strategies for the Griffiss Petroleum Spill Sites and the March 31, 2011, meeting was held to discuss planned approaches to achieve eventual site closures for the Griffiss CERCLA Sites.

March 30, 2011 – AECOM, CAPE, FPM, AFCEC, and NYSDEC

For the petroleum spill sites, LTM will continue in conjunction with additional groundwater and soil remediation through biosparging, bioventing, and oxidant injections. Remediation will be conducted at all sites except for SS070 (LTM only), and all sites are anticipated to achieve site and spill closure by the end of the period of performance (POP) except for SS054 and SS068.

March 31, 2011 – AECOM, CAPE, FPM, AFCEC, USEPA, and NYSDEC

For the CERCLA AOCs, LTM will continue at the Creek AOCs, Landfill AOCs, and SS060 (On-Base Groundwater AOCs). Groundwater remediation is also anticipated for SS060 via the use of emulsified vegetable oil injections. It is anticipated that SS060 will be closed with restricted use. LTM optimization is anticipated for the Landfill AOCs, and site closure is anticipated for the Creek AOCs. The site objectives will be supported through addition LTM.

Vapor sampling will be performed at the SD052 SVI system sites to monitor the effectiveness of the SVI systems during the POP. System and sampling optimization are the objectives at these systems.

QAPP Worksheet #10 – Problem Definition

Problem Definition

As a result of the various national defense missions carried out at the former Griffiss AFB since 1942, hazardous and toxic substances were used and hazardous wastes were generated, stored, or disposed of at various sites on the installation. The defense missions involved, among others, procurement, storage, maintenance, and shipping of war material; research and development; and aircraft O&M.

Numerous studies, investigations, and remedial actions under the U.S. Department of Defense (DOD) Installation Restoration Program (IRP) have been performed to locate, assess, quantify, and remove contaminant sources at the past toxic and hazardous waste storage, disposal, and spill sites. Pursuant to Section 105 of CERCLA, Griffiss AFB was included on the National Priorities List (NPL) on July 15, 1987. On March 20, 2009, 2,897.2 acres were deleted from the NPL. On August 21, 1990, the Air Force, USEPA, and NYSDEC entered into a Federal Facilities Agreement (FFA) under Section 120 of CERCLA.

Starting in 2002, LTM was implemented at the former Griffiss AFB. LTM is currently conducted at several petroleum spill sites, including: petroleum source removal AOCs, on-base groundwater AOCs, landfill AOCs, and creek AOCs.

Groundwater and surface water monitoring is conducted at five landfill AOCs for landfill leachate indicators and volatile organic compounds (VOCs).

Groundwater sampling is conducted at four petroleum spill sites for VOCs and at one petroleum spill site for SVOCs. Surface water will be sampled at one site for VOCs. One of the petroleum spill sites is associated with permanent biosparging systems.

Soil Vapor Intrusion (SVI) sampling is conducted at four SVI mitigation sites. Sampling consists of indoor, outdoor, and sub-slab vapor sampling for VOCs at each site.

SVI sampling is conducted at one Soil Vapor Extraction (SVE) system site. Sampling consists of indoor, outdoor, and sub-slab vapor sampling for VOCs.

Soil sampling will be conducted at one petroleum spill site for VOCs and SVOCs.

Project Description

Additional LTM for groundwater will be conducted at the petroleum spill sites, on-base groundwater AOC, and landfill AOCs. These

QAPP Worksheet #10 – Problem Definition

sites and site objectives are provided in Table 1-1.

Groundwater:

- Analysis will include VOCs for quarterly, semi-annual, and annual monitoring performed at the petroleum spill sites (SS054, SS064, and SS067) at sampling locations illustrated in Figures 17-9 through 17-11.
- Analysis will include VOCs and landfill leachate indicators (including anions, Total Kjehldahl nitrogen (TKN), ammonia, COD, Biological Oxygen Demand (BOD), TOC, TDS, alkalinity, hardness, and color) for annual monitoring performed at the five Landfill AOCs. The sampling locations are illustrated on Figures 17-2 through 17-6.
- Analysis will include VOCs and SVOCs for performance monitoring at petroleum spill site Building 785 Pipeline (sampling locations illustrated in Figure 17-12).

Surface Water:

- Analysis of VOCs for quarterly monitoring at one petroleum spill site at three locations [SS064 (Figure 17-11)]. Analysis of VOCs and landfill leachate indicators for annual monitoring at two Landfill AOCs. Three sampling locations are present at LF001 (Figure 17-2) and six sampling locations are present at LF009 (Figure 17-6). Analysis of landfill leachate indicators will be conducted for annual sampling at three Landfill AOCs (LF002, LF003, and LF007). The sampling locations are illustrated on Figures 17-3, 17-4, and 17-5.

SVI Sampling:

- One indoor air sample, one outdoor air sample, and three sub-slab vapor samples will be collected semi-annually at each SVI mitigation system. The samples will be analyzed for VOCs. The vapor sampling locations are illustrated on Figures 17-7 and 17-8.
- Two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected quarterly at ST006 Building 101 AOC. The samples will be analyzed for VOCs. The vapor sampling locations are illustrated on Figure 17-13.

Soil:

QAPP Worksheet #10 – Problem Definition

- Sampling will be conducted at Building 785 Pipeline (2 soil sampling locations). The sampling locations are illustrated in Figures 17-12.

Project Decision Condition:

In order to achieve the goals stated in the project description, the following data inputs have been identified:

- The confirmation and delineation of contamination at petroleum spill sites and AOCs throughout the former Griffiss AFB.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? Data will be used by USEPA, NYSDEC, AFCEC, CAPE, FPM, and AECOM.

What will the data be used for?

- Groundwater analytical results will be used to assess groundwater contamination data trends and to support site closure or monitoring optimization recommendations
- Surface water analytical results will be used to assess contamination data trends and the potential impacts from upgradient sites, and to support site closure or monitoring optimization recommendations
- Indoor air, outdoor air, and sub-slab vapor analytical results will be used to assess the performance of the SVI mitigation systems and the performance of the SVE system.
- Soil analytical results will be used to quantify residual contamination at the petroleum site.

What types of data are needed?

- Groundwater samples analyzed for VOCs, SVOCs, and landfill leachate indicators
- Surface water samples analyzed for VOCs and landfill leachate indicators
- Soil samples analyzed for VOCs and SVOCs
- Indoor, outdoor, and sub-slab vapor samples analyzed for VOCs.

How much data are needed?

Landfill AOCs (LF001, LF002, LF003, LF007, and LF009) – Groundwater and surface water sampling will be required until 2040. Annual monitoring rounds will be used to support any optimization recommendations.

SVI mitigation system sites (SD052 SVI systems at Buildings 774, 776, 785, and 786) – Indoor, outdoor, and sub-slab vapor samples will be required as long as the systems are in operation. The semi-annual sampling will be used to assess the effectiveness of the SVI mitigation systems.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Petroleum spill sites (SS054, SS064, and SS067) – At least four quarterly sampling rounds of data is required to determine contamination trends or to support site closure. Data will include groundwater and surface water sampling results. One monitoring event will be conducted at the Building 785 Pipeline site to assess remedial action. SVE system (ST006 Building 101) – two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected. The quarterly sampling will be used to assess the effectiveness of the SVE system.
How good does the data need to be? All analytical data will be generated from groundwater, surface water, vapor, and soil samples sent to TA. Samples will be duplicated in the field at a rate of 10% and analyzed by TA to assess sampling precision. Matrix spike/ matrix spike duplicates (MS/MSD) will be collected at a rate of 5%. Additional QC/ quality assurance (QA) protocols (field and lab) are provided in Worksheets # 20 and #22 through #33.
When will data be collected? Please refer to the CERCLA Sites and Petroleum Spill Sites Optimization Plans for this information.

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Groundwater and soil	Full data verification and validation criteria are listed in Table 12-3			
Analytical Group	VOCs, SVOCs				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ Standard Operating Procedure (SOP)[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, No. 3, and No. 4	EPA 8260B/ DV-MS-0010 EPA 8270D/ DV-MS-0012	Precision – Lab	Relative Percent Difference (RPD) < 30%	MS/MSD and/or Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x Reporting Limit (RL, considered equivalent to the limit of quantitation [LOQ]), then 30% RPD for aqueous, 50% soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1, 12-2, and 12-3	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, No. 3, and No. 4 (continued)	EPA 8260B/ DV-MS-0010 EPA 8270D/ DV-MS-0012 (continued)	Quantitation Limit	Limit of Quantitation (LOQ) > Limit of Detection (LOD) LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the Detection Limit (DL).	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	90 and 95% for soil and groundwater, respectively	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S
		External Contamination of Samples	Detections < RL	Ambient Blank	S
		Sample Contamination check during transport	Detections < LOQ	Trip Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Groundwater	Full data verification and validation criteria are listed in Table 12-6			
Analytical Group	Wet chemistry analytes				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, and No. 3	SW9056A, EPA 351.2, 350.1, 410.4, SM5210B, SM2340C, SM2120B, SW9060A, SW9066, SW9012B, SM2540C, SM2320B DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x RL (considered equivalent to the LOQ), then 20% RPD for aqueous samples, 30% for soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Table 12-4	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, and No. 3 (continued)	SW9056A, EPA 351.2, 350.1, 410.4, SM5210B, SM2340C, SM2120B, SW9060A, SW9066, SW9012B, SM2540C, SM2320B DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060 (continued)	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	> 95% laboratory analysis	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Soil gas	Full data verification and validation criteria are listed in Table 12-7			
Analytical Group	VOCs				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOP No. 6	TO-15 BR-AT-004	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x RL (considered equivalent to the LOQ), then 25% RPD. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Table 12-5	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOP No. 6 (continued)	TO-15 (continued) BR-AT-004	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ at the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	> 95% laboratory analysis	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Background information and historic levels of petroleum impact at the site.	Documents pertaining to past work at the former Griffiss AFB petroleum spill sites and AOCs.	Collection of groundwater, surface water, soil, soil gas, indoor vapor, outdoor vapor, and sub-slab vapor samples.	Data will be used to make project decisions and determine if closure requirements and clean-up goals are met.	None

QAPP Worksheet #14 – Summary of Project Tasks

Sampling Tasks:

General

1. Landfill AOCs:
   - Sampling locations are shown on Figures 17-2 through 17-6. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
   - Groundwater sampling to assess the contaminant absence/presence and data trends.
   - Surface water sampling to assess if groundwater contamination is migrating to creek environments.
2. SVI Mitigation System Sites:
   - Sampling locations are shown on Figures 17-7 and 17-8. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
   - Indoor air sampling to evaluate the effectiveness of the SVI mitigation systems and assess data trends.
   - Outdoor air sampling to be used as a reference for indoor sampling results.
   - Sub-slab vapor sampling to evaluate the effectiveness of the SVI mitigation systems and assess data trends.
3. Petroleum Spill Sites:
   - Sampling locations are shown on Figures 17-9 through 17-12. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
   - Groundwater sampling to evaluate contamination trends and the effectiveness of the groundwater remediation systems.
5. SVE System Sites:
   - Sampling locations are shown on Figure 17-13. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
   - Sub-slab vapor sampling to evaluate the effectiveness of the SVE system and assess data trends.

Samples will be collected using the SOPs attached as Appendix A of this UFP-QAPP.

QAPP Worksheet #14 – Summary of Project Tasks

Analysis Tasks:

1. Landfill AOCs
   - TA will analyze groundwater samples for VOCs using USEPA Method SW8260B (AFCEE QAPP 4.0 List), metals using USEPA Method SW6010, and Landfill leachate indicators using USEPA methods SW9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW9060A [total organic carbon (TOC)], SM2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW9066 (phenols), SW9012B (cyanide), and SW 6010B (metals including boron).
2. SVI Mitigation System Sites
   - TA will analyze vapor samples for VOCs using Method TO-15 (AFCEE QAPP 4.0 List).
3. Petroleum Spill Sites.
   - TA will analyze groundwater, surface water, and soil samples for VOCs using USEPA Method SW8260B (AFCEE QAPP 4.0 List and STARS List) and SVOCs using USEPA Method SW8270 (AFCEE QAPP 4.0 List).
4. SVE System Site
   - TA will analyze vapor samples for VOCs using Method TO-15 (AFCEE QAPP 4.0 List).

Quality Control Tasks:

1. MS/MSDs will be collected at an approximate frequency of 5%.
2. Duplicates will be collected at a rate of 10% and analyzed by TA to assess field and laboratory precision.
3. Trip blank samples will be included in each cooler containing samples for VOC analysis.
4. Ambient blanks will be collected each day that VOC samples are collected.
5. Equipment blanks will be collected from each type of non-disposable, decontaminated sampling device.

QAPP Worksheet #14 – Summary of Project Tasks

Secondary Data:

Previously collected data will be evaluated. See Worksheet #13.

Data Management Tasks:

Data will be delivered in an Environmental Restoration Program Information Management System (ERPIMS) database compatible format after data verification/ validation have been performed and data qualifiers have been added.

Documentation and Records:

1. All field documentation will be recorded in indelible ink in bound field books. These will summarize all daily field activities, weather conditions, personnel present, visitors, etc. All samples collected will be documented as to their location, which will be measured from the closest two perpendicular walls. Each day's samples and associated field measurements shall be recorded on field sampling forms. Chain of Custody (COC) forms, airbills, and sample logs will be prepared and retained for each sample.

2. A copy of the final UFP QAPP will be retained in central project file (electronically on a server) and in print form in the onsite office.

Data Packages:

TA will complete analytical data packages in accordance with the AFCEC approved forms or similar and will provide ERPIMS X file.

Assessment / Audit Tasks:

Field Sample Collection and Documentation Audits: to be determined.

Data Review Tasks

1. For the samples, TA will verify that all data are complete for samples received. All data package deliverable requirements will be met. Data will be 100% verified by FPM in accordance with this UFP-QAPP. A data verification report will be prepared for each lab work order (lab data package).

2. Verified and validated data and all related field logbooks/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required Action Limits. CA will be initiated as necessary. Final data are placed in the ERPIMS database.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,1,1,2-Tetrachloroethane	630-20-6	5	0.17	0.2	1.0
1,1,1-Trichloroethane	71-55-6	5	0.16	0.2	1.0
1,1,2,2-Tetrachloroethane	79-34-5	5	0.20	0.4	1.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	0.79	2.8	3.0
1,1,2-Trichloroethane	79-00-5	1	0.32	0.4	1.0
1,1-Dichloroethane	75-34-3	5	0.16	0.2	1.0
1,1-Dichloroethene	75-35-4	5	0.14	0.2	1.0
1,1-Dichloropropene	563-58-6	5	0.15	0.4	1.0
1,2,3-Trichlorobenzene	87-61-6	5	0.18	0.4	1.0
1,2,3-Trichloropropane	96-18-4	0.04	0.77	0.8	3.0
1,2,4-Trichlorobenzene	120-82-1	5	0.32	0.8	1.0
1,2,4-Trimethylbenzene	95-63-6	5	0.14	0.2	1.0
1,2-Dibromo-3-Chloropropane	96-12-8	0.04	0.81	1.6	5.0
1,2-Dichlorobenzene	95-50-1	3	0.13	0.2	1.0
1,2-Dichloroethane	107-06-2	0.6	0.13	0.2	1.0
1,2-Dichloropropane	78-87-5	1	0.13	0.2	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,3,5-Trimethylbenzene	108-67-8	5	0.14	0.4	1.0
1,3-Dichlorobenzene	541-73-1	3	0.16	0.2	1.0
1,3-Dichloropropane	142-28-9	5	0.15	0.2	1.0
1,4-Dichlorobenzene	106-46-7	3	0.16	0.4	1.0
1,4-Dioxane	123-91-1	6.7[3]	71	80	220
1-Chlorohexane	544-10-5	NA	0.17	0.2	1.0
2,2-Dichloropropane	594-20-7	5	0.20	0.4	1.0
2-Butanone (MEK)	78-93-3	49,000[3]	1.83	3.2	6.0
2-Hexanone	591-78-6	50	1.4	3.2	5.0
4-Chlorotoluene	106-43-4	5	0.17	0.4	1.0
4-Methyl-2-pentanone (MIBK)	108-10-1	10,000[3]	1.04	3.2	5.0
Acetone	67-64-1	50	1.9	6.4	10
Benzene	71-43-2	1	0.16	0.2	1.0
Bromobenzene	108-86-1	5	0.17	0.2	1.0
Bromochloromethane	74-97-5	5	0.10	0.2	1.0
Bromodichloromethane	75-27-4	50	0.17	0.2	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Bromoform	75-25-2	50	0.19	0.4	1.0
Bromomethane	74-83-9	5	0.21	0.4	2.0
Carbon disulfide	75-15-0	15,000[3]	0.45	0.8	2.0
Carbon tetrachloride	56-23-5	5	0.19	0.4	2.0
Chlorobenzene	108-90-7	5	0.17	0.2	1.0
Chlorobromomethane	74-97-5	830[3]	0.10	0.2	1.0
Chlorodibromomethane	124-48-1	50	0.17	0.4	1.0
Chloroethane	75-00-3	5	0.41	1.6	2.0
Chloroform	67-66-3	7	0.16	0.2	1.0
Chloromethane	74-87-3	1,900[3]	0.30	0.8	2.0
cis-1,2-Dichloroethene	156-59-2	5	0.15	0.2	1.0
cis-1,3-Dichloropropene	10061-01-5	5	0.16	0.2	1.0
Cyclohexane	110-82-7	130,000[3]	0.28	0.4	2.0
Dibromomethane	74-95-3	5	0.17	0.4	1.0
Dichlorodifluoromethane	75-71-8	5	0.31	0.8	2.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Ethylbenzene	100-41-4	5	0.16	0.2	1.0
Hexachlorobutadiene	87-68-3	0.5	0.36	0.4	1.0
Isopropylbenzene	98-82-8	5	0.19	0.4	1.0
Methyl acetate	79-20-9	160,000[3]	1.64	2	5.0
Methyl tert-butyl ether	1634-04-4	10	0.25	0.4	5.0
Methylcyclohexane	108-87-2	NA	0.36	0.4	2.0
Methylene Chloride	75-09-2	5	0.32	0.4	5.0
m-Xylene & p-Xylene	179601-23-1	5	0.34	0.8	2.0
Naphthalene	91-20-3	10	0.22	0.8	1.0
n-Butylbenzene	104-51-8	5	0.32	0.4	1.0
N-Propylbenzene	103-65-1	5	0.16	0.2	1.0
o-Xylene	95-47-6	5	0.19	0.4	1.0
p-Isopropyltoluene	99-87-6	5	0.17	0.4	1.0
sec-Butylbenzene	135-98-8	5	0.17	0.4	1.0
Styrene	100-42-5	5	0.17	0.4	1.0
tert-Butylbenzene	98-06-6	5	0.16	0.4	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Tetrachloroethene	127-18-4	5	0.20	0.4	1.0
Toluene	108-88-3	5	0.17	0.4	1.0
trans-1,2-Dichloroethene	156-60-5	5	0.15	0.2	1.0
trans-1,3-Dichloropropene	10061-02-6	0.4	0.19	0.4	1.0
Trichloroethene	79-01-6	5	0.16	0.2	1.0
Trichlorofluoromethane	75-69-4	5	0.29	0.8	2.0
Vinyl chloride	75-01-4	2	0.10	0.4	1.5
Xylenes, Total	1330-20-7	5	0.53	1.2	3.0

1. New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999

2 Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.

[3]EPA Regional Screening Levels Tapwater Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index

NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	19,000[4]	0.56	1.0	5.0
1,1,1-Trichloroethane	71-55-6	500,000/100,000[1]	0.52	1.0	5.0
1,1,2,2-Tetrachloroethane	79-34-5	NA/35,000[3]	0.61	1.0	5.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA/100,000[3]	0.45	20	20
1,1,2-Trichloroethane	79-00-5	11,000[4]	0.88	1.0	5.0
1,1-Dichloroethane	75-34-3	240,000/19,000[1]	0.21	0.8	5.0
1,1-Dichloroethene	75-35-4	500,000/100,000[1]	0.59	1.0	5.0
1,1-Dichloropropene	563-58-6	NA/NA	0.54	1.0	5.0
1,2,3-Trichlorobenzene	87-61-6	490,000[4]	0.75	1.0	5.0
1,2,3-Trichloropropane	96-18-4	NA/80,000[3]	0.81	1.0	5.0
1,2,4-Trichlorobenzene	120-82-1	220,000[4]	0.73	1.0	5.0
1,2,4-Trimethylbenzene	95-63-6	190,000/47,000[1]	0.58	1.0	5.0
1,2-Dibromo-3-Chloropropane	96-12-8	54[4]	0.60	1.0	10
1,2-Dichlorobenzene	95-50-1	500,000/100,000[1]	0.45	1.0	5.0
1,2-Dichloroethane	107-06-2	30,000/2,300[1]	0.70	1.0	5.0
1,2-Dichloropropane	78-87-5	9,400[4]	0.55	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,3,5-Trimethylbenzene	108-67-8	190,000/47,000[1]	0.57	1.0	5.0
1,3-Dichlorobenzene	541-73-1	280,000/17,000[1]	0.48	1.0	5.0
1,3-Dichloropropane	142-28-9	16,000,000[4]	0.51	1.0	5.0
1,4-Dichlorobenzene	106-46-7	130,000/9,800[1]	0.78	1.0	5.0
1,4-Dioxane	123-91-1	130,000/9,800[1]	56.1	80	500
1-Chlorohexane	544-10-5	NA/NA	0.63	0.80	5.0
2,2-Dichloropropane	594-20-7	NA/NA	0.44	1.0	5.0
2-Butanone (MEK)	78-93-3	500,000/100,000[1]	1.83	6.4	20
2-Hexanone	591-78-6	2,100,000[4]	4.89	10	20
4-Chlorotoluene	106-43-4	NA/NA	0.78	1.0	5.0
4-Methyl-2-pentanone (MIBK)	108-10-1	53,000,000[4]	4.36	10	20
Acetone	67-64-1	500,000/100,000[1]	5.38	10	20
Benzene	71-43-2	44,000/2,900[1]	0.47	1.0	5.0
Bromobenzene	108-86-1	3,000,000[4]	0.49	1.0	5.0
Bromochloromethane	74-97-5	1,600,000[4]	0.30	1.0	5.0
Bromodichloromethane	75-27-4	2,700[4]	0.22	0.8	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Bromoform	75-25-2	620,000[4]	0.23	0.8	5.0
Bromomethane	74-83-9	73,000[4]	0.50	1.0	10
Carbon disulfide	75-15-0	NA/100,000[3]	0.42	1.0	5.0
Carbon tetrachloride	56-23-5	22,000/1,400[1]	0.63	1.0	5.0
Chlorobenzene	108-90-7	500,000/100,000[1]	0.54	1.0	5.0
Chlorodibromomethane	124-48-1	NA/NA	0.57	1.0	5.0
Chloroethane	75-00-3	NA/NA	0.89	1.0	10
Chloroform	67-66-3	350,000/10,000[1]	0.29	1.0	10
Chloromethane	74-87-3	1,200,000[4]	0.77	1.0	10
cis-1,2-Dichloroethene	156-59-2	500,000/59,000[1]	0.56	1.0	5.0
cis-1,3-Dichloropropene	10061-01-5	NA/NA	1.29	2.0	5.0
Cyclohexane	110-82-7	70,000,000[4]	0.40	1.6	5.0
Dibromomethane	74-95-3	250,000[4]	0.84	1.0	5.0
Dichlorodifluoromethane	75-71-8	940,000[4]	0.52	1.0	10
Ethylbenzene	100-41-4	390,000/30,000[1]	0.67	1.0	5.0
Hexachlorobutadiene	87-68-3	62,000[4]	0.55	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Isopropylbenzene	98-82-8	NA/100,000[3]	0.59	1.0	5.0
Methyl acetate	79-20-9	780,000,000[4]	2.75	4.0	8.5
Methyl tert-butyl ether	1634-04-4	500,000/62,000[1]	0.34	1.0	20
Methylcyclohexane	108-87-2	NA/NA	0.42	0.80	5.0
Methylene Chloride	75-09-2	500,000/51,000[1]	1.6	3.2	5.0
m-Xylene & p-Xylene	179601-23-1	NA/NA	1.04	2.0	3.2
Naphthalene	91-20-3	500,000/100,000[1]	0.63	1.0	5.0
n-Butylbenzene	104-51-8	500,000/100,000[1]	0.56	1.0	5.0
n-Propylbenzene	103-65-1	500,000/100,000[1]	0.58	1.0	5.0
o-Xylene	95-47-6	6,900,000[4]	0.61	1.0	5.0
p-Isopropyltoluene	99-87-6	NA/NA	0.49	1.0	5.0
sec-Butylbenzene	135-98-8	500,000/100,000[1]	0.77	1.0	5.0
Styrene	100-42-5	100,000,000[4]	0.63	1.0	5.0
tert-Butylbenzene	98-06-6	500,000/100,000[1]	0.50	1.0	5.0
Tetrachloroethene	127-18-4	150,000/5,500[1]	0.59	1.0	5.0
Toluene	108-88-3	500,000/100,000[1]	0.69	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
trans-1,2-Dichloroethene	156-60-5	500,000/100,000[1]	0.39	1.0	5.0
trans-1,3-Dichloropropene	10061-02-6	NA/NA	0.67	1.0	5.0
Trichloroethene	79-01-6	200,000/10,000[1]	0.23	0.8	5.0
Trichlorofluoromethane	75-69-4	7,900,000[4]	1.04	2.0	10
Vinyl chloride	75-01-4	13,000/210[1]	1.34	2.0	5.0
Xylenes, Total	1330-20-7	500,000/100,000[1]	1.65	3.0	8.2

1 - NYSDEC 60 NYCRR Part 375 Restricted and UnrestrictedUse Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Unrestricted clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - CP-51/ Soil Cleanup Guidance, October 2010.
4 - EPA Regional Screening Levels Resident Soil Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,1'-Biphenyl	92-52-4	5	1.75	2.0	10
1,2,4,5-Tetrachlorobenzene	95-94-3	5	1.73	2.0	10
1,2,4-Trichlorobenzene	120-82-1	5	0.28	1.0	10
1,2-Dichlorobenzene	95-50-1	3	0.23	1.0	10
1,3-Dichlorobenzene	541-73-1	3	0.30	1.0	10
1,4-Dichlorobenzene	106-46-7	3	0.32	1.0	10
2,2'-oxybis[1-chloropropane]	108-60-1	5	0.28	1.0	10
2,3,4,6-Tetrachlorophenol	58-90-2	1,700[3]	2.0	2.0	50
2,4,5-Trichlorophenol	95-95-4	8,900[3]	0.45	1.0	20
2,4,6-Trichlorophenol	88-06-2	35[3]	0.29	1.0	20
2,4-Dichlorophenol	120-83-2	1	0.64	2.0	10
2,4-Dimethylphenol	105-67-9	1	0.58	4.0	10
2,4-Dinitrophenol	51-28-5	1	10	20	80
2,4-Dinitrotoluene	121-14-2	5	1.66	4.0	20
2,6-Dinitrotoluene	606-20-2	5	1.89	4.0	20
2-Chloroaniline	106-47-8	5	2.14	5.0	25

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
2-Chloronaphthalene	91-58-7	10	0.26	1.0	10
2-Chlorophenol	95-57-8	710[3]	2.0	4.0	10
2-Methylnaphthalene	91-57-6	270[3]	0.29	1.0	10
2-Methylphenol	95-48-7	1	0.98	4.0	10
2-Nitroaniline	88-74-4	5	1.73	4.0	50
2-Nitrophenol	88-75-5	NA	0.39	1.0	20
3,3'-Dichlorobenzidine	91-94-1	5	2.0	10	50
3-Nitroaniline	99-09-2	5	2.0	2.0	50
4,6-Dinitro-2-methylphenol	534-52-1	12[3]	4.0	10	80
4-Bromophenyl phenyl ether	101-55-3	NA	0.43	1.0	10
4-Chloro-3-methylphenol	59-50-7	NA	2.41	5.0	20
4-Chloroaniline	106-47-8	5	2.14	5.0	25
4-Chlorophenyl phenyl ether	7005-72-3	NA	1.66	4.0	10
4-Methylphenol	106-44-5	NA	0.25	1.0	20
4-Nitroaniline	100-01-6	5	2.0	4.0	50
4-Nitrophenol	100-02-7	NA	1.23	10	50

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Acenaphthene	83-32-9	20	0.28	1.0	10
Acenaphthylene	208-96-8	NA	0.49	1.0	10
Acetophenone	98-86-2	15,000[3]	0.24	5.0	10
Anthracene	120-12-7	50	0.42	1.0	10
Atrazine	1912-24-9	7.5	0.73	10	50
Benzaldehyde	100-52-7	15,000[3]	2.0	2.0	10
Benzo[a]anthracene	56-55-3	0.002[4*]	0.35	1.0	10
Benzo[a]pyrene	50-32-8	Not Detectable	0.31	1.0	10
Benzo[b]fluoranthene	205-99-2	0.002[4*]	0.531	1.0	10
Benzo[g,h,i]perylene	191-24-2	NA	0.50	1.0	10
Benzo[k]fluoranthene	207-08-9	0.002[4*]	0.46	1.0	10
Benzoic acid	65-85-0	580,000[3]	10	50	80
Benzyl alcohol	100-51-6	15,000[3]	0.23	1.0	25
Bis(2-chloroethoxy)methane	111-91-1	5	0.97	4.0	10
Bis(2-chloroethyl)ether	111-44-4	1	0.41	1.0	20
Bis(2-ethylhexyl) phthalate	117-81-7	5	0.56	1.0	10

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Butyl benzyl phthalate	85-68-7	50	1.0	4.0	20
Caprolactam	105-60-2	77,000[3]	5.0	10	35
Carbazole	86-74-8	NA	0.43	1.0	10
Chrysene	218-01-9	0.002[4*]	0.54	1.0	10
Dibenz(a,h)anthracene	53-70-3	0.029[3*]	0.51	1.0	10
Dibenzofuran	132-64-9	58[3]	0.29	1.0	10
Diethyl phthalate	84-66-2	50	0.38	1.0	20
Dimethyl phthalate	131-11-3	50	0.21	1.0	20
Di-n-butyl phthalate	84-74-2	50	1.16	4.0	20
Di-n-octyl phthalate	117-84-0	50	0.35	1.0	20
Fluoranthene	206-44-0	50	0.20	1.0	20
Fluorene	86-73-7	50	0.31	1.0	10
Hexachlorobenzene	118-74-1	0.04*	0.66	1.0	10
Hexachlorobutadiene	87-68-3	0.5*	3.3	10	30
Hexachlorocyclopentadiene	77-47-4	5	10	20	50
Hexachloroethane	67-72-1	5	2.1	4.0	10

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Indeno[1,2,3-cd]pyrene	193-39-5[4]	0.002*	0.65	1.0	10
Isophorone	78-59-1	50	0.21	1.0	10
Naphthalene	91-20-3	10	0.29	1.0	10
Nitrobenzene	98-95-3	0.4	0.81	2.0	20
N-Nitrosodi-n-propylamine	621-64-7	0.093[3*]	0.35	1.0	20
N-Nitrosodiphenylamine	86-30-6	50[3]	0.44	1.0	10
Pentachlorophenol	87-86-5	1	20	40	80
Phenanthrene	85-01-8	50	0.26	1.0	10
Phenol	108-95-2	1	2.0	5.0	10
Pyrene	129-00-0	50	0.37	1.0	10

* - Analyte has DL greater than PA is below the DL. If the analyte is not detected, it will be considered below the standard.
1 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999
2 - Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
3 - EPA Regional Screening Levels Tapwater Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index
4 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Technical and Operational Guidance Series, Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, June 1998
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,1'-Biphenyl	92-52-4	510,000[4]	50	167	330
1,2,4,5-Tetrachlorobenzene	95-94-3	180,000[4]	49	67	330
1,2,4-Trichlorobenzene	120-82-1	220,000[4]	28	33	330
1,2-Dichlorobenzene	95-50-1	500,000/100,000[1]	22	33	330
1,3-Dichlorobenzene	541-73-1	280,000/17,000[1]	12	33	330
1,4-Dichlorobenzene	106-46-7	130,000/9,800[1]	13.6	33	330
2,2'-oxybis[1-chloropropane]	108-60-1	NA/NA	23	33	330
2,3,4,6-Tetrachlorophenol	58-90-2	18,000,000[4]	137	167	1600
2,4,5-Trichlorophenol	95-95-4	NA/100,000[3]	10	130	330
2,4,6-Trichlorophenol	88-06-2	440,000[4]	10	66	330
2,4-Dichlorophenol	120-83-2	NA/100,000[3]	10	66	330
2,4-Dimethylphenol	105-67-9	12,000,000[4]	66	130	330
2,4-Dinitrophenol	51-28-5	NA/100,000[3]	333	670	1600
2,4-Dinitrotoluene	121-14-2	16,000[4]	66	130	330
2,6-Dinitrotoluene	606-20-2	NA/1,030[3]	28	66	330
2-Chloroaniline	95-51-2	NA/NA	81.9	130	330

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
2-Chloronaphthalene	91-58-7	NA/NA	10	33	330
2-Chlorophenol	95-57-8	NA/100,000[3]	21	33	330
2-Methylnaphthalene	91-57-6	NA/410[3]	19	33	330
2-Methylphenol	95-48-7	500,000/100,000[1]	13	33	330
2-Nitroaniline	88-74-4	6,100,000[4]	50	66	1600
2-Nitrophenol	88-75-5	NA/NA	10	66	330
3,3'-Dichlorobenzidine	91-94-1	11,000[4]	90	330	1600
3-Nitroaniline	99-09-2	NA/NA	73	133	1600
4,6-Dinitro-2-methylphenol	534-52-1	49,000[4]	330	660	1600
4-Bromophenyl phenyl ether	101-55-3	NA/NA	19	33	330
4-Chloro-3-methylphenol	59-50-7	NA/NA	66	130	330
4-Chloroaniline	106-47-8	NA/100,000[3]	81.9	130	330
4-Chlorophenyl phenyl ether	7005-72-3	NA/NA	21	66	330
4-Methylphenol	106-44-5	500,000/34,000[3]	33	66	330
4-Nitroaniline	100-01-6	240,000[4]	72.5	130	1600
4-Nitrophenol	100-02-7	NA/NA	97	330	1600

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Acenaphthene	83-32-9	500,000/100,000[1]	10.3	17	330
Acenaphthylene	208-96-8	500,000/100,000[1]	17	33	330
Acetophenone	98-86-2	78,000,000[4]	20	33	330
Anthracene	120-12-7	500,000/100,000[1]	17	33	330
Atrazine	1912-24-9	21,000[4]	37	130	330
Benzaldehyde	100-52-7	78,000,000[4]	39	67	330
Benzo[a]anthracene	56-55-3	5,600/1,000[1]	20	33	330
Benzo[a]pyrene	50-32-8	1,000/1,000[1]	20	33	330
Benzo[b]fluoranthene	205-99-2	5,600/1,000[1]	26.2	33	330
Benzo[g,h,i]perylene	191-24-2	500,000/100,000[1]	16	33	330
Benzo[k]fluoranthene	207-08-9	56,000/1,000[1]	40	66	330
Benzoic acid	65-85-0	NA/100,000[3]	330	660	1600
Benzyl alcohol	100-51-6	61,000,000[4]	10	33	330
Bis(2-chloroethoxy)methane	111-91-1	1,800,000[4]	23	66	330
Bis(2-chloroethyl)ether	111-44-4	6,900[4]	16.6	33	330
Bis(2-ethylhexyl) phthalate	117-81-7	NA/50,000[3]	46	66	330

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Butyl benzyl phthalate	85-68-7	NA/100,000[3]	43	66	330
Caprolactam	105-60-2	310,000,000[4]	106	330	1600
Carbazole	86-74-8	NA/NA	36	67	330
Chrysene	218-01-9	56,000/1,000[1]	27	33	330
Dibenz(a,h)anthracene	53-70-3	560/330[1]	19	33	330
Dibenzofuran	132-64-9	350,000/14,000[1]	20	33	330
Diethyl phthalate	84-66-2	NA/100,000[3]	26	33	660
Dimethyl phthalate	131-11-3	NA/100,000[3]	23	33	330
Di-n-butyl phthalate	84-74-2	NA/100,000[3]	29	33	330
Di-n-octyl phthalate	117-84-0	100,000[3]	14.4	66	330
Fluoranthene	206-44-0	500,000/100,000[1]	36	66	330
Fluorene	86-73-7	500,000/100,000[1]	18	33	330
Hexachlorobenzene	118-74-1	6,000/330[1]	29	66	330
Hexachlorobutadiene	87-68-3	62,000[4]	10	66	330
Hexachlorocyclopentadiene	77-47-4	3,700,000[4]	50	66	1700
Hexachloroethane	67-72-1	120,000[4]	21.3	33	330

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Indeno[1,2,3-cd]pyrene	193-39-5	5,600/500[1]	22	33	330
Isophorone	78-59-1	NA/100,000[3]	17	33	330
Naphthalene	91-20-3	500,000/100,000[1]	31	66	330
Nitrobenzene	98-95-3	69,000/3,700[3]	22	33	330
N-Nitrosodi-n-propylamine	621-64-7	690[4]	31	66	330
N-Nitrosodiphenylamine	86-30-6	990,000[4]	21	33	330
Pentachlorophenol	87-86-5	6,700/2,400[1]	330	670	1600
Phenanthrene	85-01-8	500,000/100,000[1]	17	33	330
Phenol	108-95-2	500,000/100,000[1]	18	33	330
Pyrene	129-00-0	500,000/100,000[1]	12.1	33	400

1 - NYSDEC 60 NYCRR Part 375 Restricted and UnrestrictedUse Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Residential clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - CP-51/ Soil Cleanup Guidance, October 2010.
4 - EPA Regional Screening Levels Resident Soil Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Landfill Leachate Indicators (SW9056, 351.2, 350.2, 410.4, 405.1, SW9060, SM2540C, SM2320B, 130.2, 110.2, SW9066, SW9012, and SW6010B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Achievable Laboratory Limits[1]		
			DL (mg/L)	LOD (mg/L)	LOQ (mg/L)
Bromide	24959-67-9	2[4]	0.113	0.20	0.5
Chloride	16887-00-6	250[4]	0.254	0.50	3
Fluoride	16984-48-8	1.5	0.06	0.10	1
Nitrate as N	14797-55-8	10	0.042	0.10	0.5
Nitrate + Nitrite as N	STL00217	10	0.042	0.10	0.5
Nitrite as N	14797-65-0	1	0.049	0.10	0.5
Orthophosphate as P	14265-44-2	NA	0.187	NA	0.5
Sulfate	14808-79-8	250	0.232	0.50	5
Total Organic Carbon	7440-44-0	NA	0.155	0.40	1.0
Total Phenols	64743-03-9	NA	0.009	0.005	0.02
Cyanide	57-12-5	0.2	0.002	0.002	0.010
Total Dissolved Solids	STL00242	500	4.70	10	10
Hardness	STL00009	NA	1.3	1.5	5.0
Alkalinity	STL00171	NA	1.07	2.0	5.0
BOD	STL00311	NA	0.236	0.6	2.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Landfill Leachate Indicators (SW9056, 351.2, 350.2, 410.4, 405.1, SW9060, SM2540C, SM2320B, 130.2, 110.2, SW9066, SW9012, and SW6010B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Achievable Laboratory Limits[1]		
			DL (mg/L)	LOD (mg/L)	LOQ (mg/L)
Total Kjeldahl Nitrogen (TKN)	STL00296	1	0.18	0.5	1.0
Color	STL00153	15	N/A	5 pcu	5 pcu
COD	STL00070	NA	4.06	10	20

1 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999
2 - Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
3 - EPA Regional Screening Levels Tap-water Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index
4 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Technical and Operational Guidance Series, Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, June 1998
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD ($\mu g/m^3$)	LOQ ($\mu g/m^3$)
Dichlorodifluoromethane	75-71-8	NA	0.099	0.64	2.5
Freon 22	75-45-6	NA	0.081	0.46	1.8
1,2-Dichlorotetrafluoroethane	76-14-2	NA	0.14	0.56	1.4
Chloromethane	74-87-3	818/263	0.070	0.08	1.0
n-Butane	106-97-8	NA	0.052	0.1	1.2
Vinyl chloride	75-01-4	NA	0.023	0.2	0.51
1,3-Butadiene	106-99-0	NA	0.055	0.09	0.44
Bromomethane	74-83-9	NA	0.10	0.16	0.78
Chloroethane	75-00-3	NA	0.087	0.11	1.3
Bromoethene(Vinyl Bromide)	593-60-2	NA	0.083	0.17	0.87

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Trichlorofluoromethane	75-69-4	NA	0.12	0.73	1.1
Freon TF	76-13-1	NA	0.15	0.31	1.5
1,1-Dichloroethene	75-35-4	NA	0.34	0.32	0.79
Acetone	67-64-1	NA	0.95	0.31	12
Isopropyl alcohol	67-63-0	NA	0.19	0.32	12
Carbon disulfide	75-15-0	20,440/2,440	0.062	0.62	1.6
3-Chloropropene	107-05-1	NA	0.15	0.13	1.6
Methylene Chloride	75-09-2	1,740/1,740	0.080	0.14	1.7
tert-Butyl alcohol	75-65-0	NA	0.12	0.61	15
Methyl tert-butyl ether	1634-04-4	87,600/8,760	0.054	0.14	0.72
trans-1,2-Dichloroethene	156-60-5	NA	0.091	0.32	0.79
n-Hexane	110-54-3	20,440/2,044	0.070	0.28	0.70

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
1,1-Dichloroethane	75-34-3	NA	0.093	0.53	0.81
Methyl Ethyl Ketone	78-93-3	146,000/14,600	0.074	0.12	1.5
cis-1,2-Dichloroethene	156-59-2	1,022/102	0.33	0.16	0.79
1,2-Dichloroethene, Total	540-59-0	NA	0.091	0.16	0.79
Chloroform	67-66-3	36/36	0.12	0.39	0.98
Tetrahydrofuran	109-99-9	NA	0.086	0.12	15
1,1,1-Trichloroethane	71-55-6	146,000/14,600	0.11	0.71	1.1
Cyclohexane	110-82-7	175,200/17,520	0.065	0.45	0.69
Carbon tetrachloride	56-23-5	55/55	0.082	0.82	1.3
2,2,4-Trimethylpentane	540-84-1	NA	0.070	0.61	0.93
Benzene	71-43-2	105/88	0.058	0.13	0.64
1,2-Dichloroethane	107-06-2	31/31	0.073	0.32	0.81

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
n-Heptane	142-82-5	NA	0.070	0.16	0.82
Trichloroethene	79-01-6	409/41	0.049	0.43	1.1
Methyl methacrylate	80-62-6	NA	0.066	0.16	2.0
1,2-Dichloropropane	78-87-5	NA	0.11	0.18	0.92
1,4-Dioxane	123-91-1	NA	0.25	1.2	18
Bromodichloromethane	75-27-4	NA	0.080	0.54	1.3
cis-1,3-Dichloropropene	10061-01-5	NA	0.059	0.18	0.91
methyl isobutyl ketone	108-10-1	87,600/8,760	0.14	0.33	2.0
Toluene	108-88-3	146,000/14,600	0.053	0.15	0.75
trans-1,3-Dichloropropene	10061-02-6	NA	0.068	0.09	0.91
1,1,2-Trichloroethane	79-00-5	NA	0.087	0.22	1.1
Tetrachloroethene	127-18-4	139/102	0.10	0.27	1.4

QAPP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Methyl Butyl Ketone (2-Hexanone)	591-78-6	NA	0.16	0.53	2.0
Dibromochloromethane	124-48-1	NA	0.094	0.68	1.7
1,2-Dibromoethane	106-93-4	NA	0.11	0.31	1.5
Chlorobenzene	108-90-7	NA	0.060	0.37	0.92
Ethylbenzene	100-41-4	743/743	0.065	0.35	0.87
m,p-Xylene	179601-23-1	2,920/292	0.096	0.69	2.2
Xylene, o-	95-47-6	2,920/292	0.069	0.35	0.87
Xylene (total)	1330-20-7	2,920/292	0.069	0.35	0.87
Styrene	100-42-5	29,200/2,920	0.047	0.34	0.85
Bromoform	75-25-2	NA	0.074	0.41	2.1

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Cumene	98-82-8	NA	0.054	0.39	0.98
1,1,2,2-Tetrachloroethane	79-34-5	NA	0.076	0.89	1.4
n-Propylbenzene	103-65-1	NA	0.064	0.98	0.98
4-Ethyltoluene	622-96-8	NA	0.074	0.64	0.98
1,3,5-Trimethylbenzene	108-67-8	175/18	0.093	0.64	0.98
2-Chlorotoluene	95-49-8	NA	0.067	0.67	1.0
tert-Butylbenzene	98-06-6	NA	0.060	0.71	1.1
1,2,4-Trimethylbenzene	95-63-6	175/18	0.10	0.64	0.98
sec-Butylbenzene	135-98-8	NA	0.082	0.71	1.1
4-Isopropyltoluene	99-87-6	NA	0.11	0.71	1.1
1,3-Dichlorobenzene	541-73-1	3,212/321	0.11	0.78	1.2

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
1,4-Dichlorobenzene	106-46-7	23,360/2,336	0.11	0.78	1.2
Benzyl chloride	100-44-7	NA	0.11	0.67	1.0
n-Butylbenzene	104-51-8	NA	0.12	0.71	1.1
1,2-Dichlorobenzene	95-50-1	31/31	0.16	0.78	1.2
1,2,4-Trichlorobenzene	120-82-1	NA	0.22	0.96	3.7
Hexachlorobutadiene	87-68-3	NA	0.31	2.1	2.1
Naphthalene	91-20-3	NA	0.20	1.7	2.6

[1]Air Force calculated sub-slab screening values/indoor screening values.
[2] Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #16 – Project Schedule / Timeline Table

The project schedule is provided in Appendix C.

QAPP Worksheet #17 – Sampling Design and Rationale

Sampling Approach:

The former Griffiss AFB site location map is provided on Figure 17-1. Sample locations for the Landfill AOCs, SVI Mitigation sites, SVE System Site, and Petroleum Spill Sites are illustrated in Figures 17-2 through 17-13.

Activities at the sites listed above will include the collection of groundwater, surface water, indoor air, outdoor air, sub-slab vapor, soil gas, and soil sampling.

Sampling Design and Rationale

Additional LTM for groundwater will be conducted at the petroleum spill sites, and landfill AOCs. Groundwater samples will be collected at existing monitoring wells at the Landfill AOCs (Figures 17-2 through 17-6). Groundwater samples will also be collected at existing monitoring wells and newly installed monitoring wells at the petroleum spill sites (Figures 17-9 through 17-13). Collection of the groundwater samples will be conducted using the low flow sampling and bailer sampling methods. The sampling will be conducted at a network of monitoring wells that achieves the optimal coverage of the potential and residual groundwater contamination. All field parameters collected during sampling (Appendix A) will also be used to assess and document groundwater flow and water quality parameters. The groundwater flow will also be used to generate contamination plume figures provided in the future LTM Reports

Surface water sampling will be conducted at one petroleum spill site, and five landfill AOCs. Surface water will be collected at three locations at the Apron 2 petroleum spill site (Figure 17-11). Samples will be analyzed for VOCs. Surface water will also be collected at six locations for Landfill 6 (including two leachate sampling locations), three locations for Landfill 1, 2/3, and 5, and two locations for Landfill 7. Sample analysis includes VOCs, and landfill leachate indicators. Samples will be collected using the grab sampling method. The sample results will be used to confirm contamination data trends and to delineate the potential impacts from upgradient sites.

Two indoor air samples, one outdoor air sample, and three sub-slab vapor sample will be collected semi-annually at each SVI mitigation system (Figures 17-7 and 17-8). The samples will be analyzed for VOCs to evaluate the effectiveness of the SVI mitigation systems in mitigating the potential for SVI.

Two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected quarterly at the SVE system associated with ST006 Building 101 AOC (Figure 17-13). The samples will be analyzed for VOCs.

Soil sampling will be conducted to confirm the levels of residual contamination following the remedial action at the Building 785 Pipeline site

QAPP Worksheet #17 – Sampling Design and Rationale

(Figure 17-12). The samples will be analyzed for VOCs and SVOCs.

All field parameter measurements will be documented in the daily chemical QC reports issued as part of the LTM/Sampling Reports. At monitoring wells, the groundwater elevations will be used to delineate the contaminant plumes and groundwater flows for figure product and recommendation support.

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
LF001 (Landfill 1)	Groundwater	0 - 30	VOCs	Low-to-Medium	35	SOP No. 2	See Worksheet #17
LF001 (Landfill 1)	Groundwater	0 - 30	Landfill leachate indicators	Low-to-Medium	55	SOP No. 2	See Worksheet #17
LF001 (Landfill 1)	Surface Water	N/A	VOCs/Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF002 (Landfill 2/3)	Groundwater	5 - 35	Landfill leachate indicators	Low-to-Medium	20	SOP No. 2	See Worksheet #17
LF002 (Landfill 2/3)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF003 (Landfill 7)	Groundwater	20 - 30	Landfill leachate indicators	Low-to-Medium	20	SOP No. 2	See Worksheet #17
LF003 (Landfill 7)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	6	SOP No. 3	See Worksheet #17
LF007 (Landfill 5)	Groundwater	15 - 20	Landfill leachate indicators	Low-to-Medium	15	SOP No. 2	See Worksheet #17
LF007 (Landfill 5)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	9	SOP No. 3	See Worksheet #17
LF009 (Landfill 6)	Groundwater	0 - 100	VOCs	Low-to-Medium	35	SOP No. 2	See Worksheet #17
LF009 (Landfill 6)	Groundwater	0 - 100	Landfill leachate indicators	Low-to-Medium	95	SOP No. 2	See Worksheet #17

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
LF009 (Landfill 6)	Surface Water	N/A	VOCs /Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF009 (Landfill 6)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
SD052 (SVI Systems -Building 774)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 776)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 785)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 786)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SS054 (Building 781)	Groundwater	40 - 60	VOCs	Medium-to-High	170	SOP No. 2	See Worksheet #17
SS064 (Apron 2)	Groundwater	20 - 30	VOCs	Medium-to-High	84	SOP No. 2	See Worksheet #17

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
SS064 (Apron 2)	Surface water	0 - 1	VOCs	Low	48	SOP No. 3	See Worksheet #17
SS068 (Building 789)	Groundwater	20 - 30	VOCs	Medium-to-High	120	SOP No. 2	See Worksheet #17
ST006 Building 101 AOC	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low	40	SOP No. 6	See Worksheet #17
Building 785 Pipeline	Groundwater	20 - 25	VOCs and SVOCs	Low	4	SOP No. 2	See Worksheet #17
Building 785 Pipeline	Soil	0 - 25	VOCs and SVOCs	Low	4	SOP No. 1	See Worksheet #17

[1]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Preparation and Analytical Method / SOP Reference[1]	Sample Volume	Containers	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation, analysis)
Water	General Chemistry	Low	DV-WC-0006	1, 250mL amber glass jar	100 mL	H_2SO_4, pH < 2; Cool < 6°C	28 days
Water	General Chemistry	Low	DV-WC-0040	1 Liter amber glass jar	1000 mL	H_2SO_4, pH < 2; Cool < 6°C	28 days
Water	General Chemistry	Low	DV-WC-0020	1, 50mL, HDPE	15 mL	Cool < 6°C	48 Hours (NO_2, NO_3, & OPO_4) / 28 Days (Fl, Cl, Br, SO_4)
Water	General Chemistry	Low	DV-WC-0082	1, 250mL, HDPE	100 mL	NaOH, pH >12; Cool < 6°C	14 Days
Water	General Chemistry	Low	DV-WC-0060	One 100mL, HDPE	25 mL	HNO_3, pH < 2; Cool < 6°C	180 days
Water	MS VOA	Low-to-Medium	DV-MS-0010	Three 40mL glass VOA Vials	40 ml glass VOA vial	<6°C; adjust pH <2; 0.008% Na2S2O3[4]	14 days, Preserved; 7 days, Unpreserved
Soil	MS VOA	Low-to-Medium	DV-MS-0010	Two 25g EnCore™	50 grams	DI water/frozen or Methanol; <6°C	14 days, Preserved; 7 days, Unpreserved

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Preparation and Analytical Method / SOP Reference[1]	Sample Volume	Containers	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation, analysis)
Water	MS Semi VOA	Low	DV-MS-0012 / DV-OP-0006, DV-OP-0007, & DV-OP-0008	Two 1 liter, amber	2000 mL	Cool < 6°C	7 days to extract; 40 days from extract
Soil	MS Semi VOA	Low	DV-MS-0012 / DV-OP-0007 & DV-OP-0010	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract
Soil	GC Semi VOA	Low	DV-GC-0020 & DV-GC-0026 / DV-OP-0007	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract
Soil	GC Semi VOA	Low	DV-GC-0021 & DV-GC-0030 / DV-OP-0007	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract
Soil Vapor	VOCs	Low-to-Medium	TO-15/005(BR-AT-004)	200 mL	6 Liter Summa Canister	NA	30 days

[1] Refer to the Analytical SOP References table (Worksheet #23).
[2] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
[3] The minimum sample size is based on analysis allowing for sufficient sample for reanalysis. Additional volume is needed for the laboratory Matrix Spike/Matrix Spike Duplicate sample analysis.
[4] Free Chlorine must be removed by the appropriate addition of Na2S2O3. This preservation is not necessary if free chlorine is not present in the groundwater.
[5] If hydrocarbons within the boiling point range of nC6 and nC12 are suspected, soil samples should be collected in pre-weighed VOA vials with PFTE lined caps.

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
LF001 (Landfill 1)	Groundwater	VOCs	Low-to-Medium	DV-MS-0010	28	2	1	4	36
LF001 (Landfill 1)	Groundwater	Landfill leachate indicators	Low-to-Medium	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	62	6	3	0	71
LF001 (Landfill 1)	Surface Water	VOCs /Landfill leachate indicators	Low	DV-MS-0010, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	0	4	20
LF002 (Landfill 2/3)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	18	1- 2	1	0	21
LF002 (Landfill 2/3)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 - 1	1	0	11
				DV-WC-0006,					

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
LF003 (Landfill 7)	Groundwater	Landfill leachate indicators	Low	DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	24	2	1	0	27
LF003 (Landfill 7)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 – 1	0	0	10
LF007 (Landfill 5)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	1	0	17
LF007 (Landfill 5)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 - 1	0	0	10
LF009 (Landfill 6)	Groundwater	VOCs	Low-to-Medium	DV-MS-0010	28	1 – 2	1	4	35
LF009 (Landfill 6)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	76	7 - 8	3	0	87

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
LF009 (Landfill 6)	Surface Water	VOCs/Landfill leachate indicators	Low	DV-MS-0010, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 – 1	0	4	20
LF009 (Landfill 6)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	0	0	16
SD052 (SVI Systems - Building 774)	SVI Vapor	VOCs	Low	BR-AT-004	22	2	1	5	30
SD052 (SVI Systems - Building 776)	SVI Vapor	VOCs	Low	BR-AT-004	22	2	1	5	30
SD052 (SVI Systems - Building 785)	SVI Vapor	VOCs	Low-to-Medium	BR-AT-004	22	2	1	5	30
SD052 (SVI Systems - Building 786)	SVI Vapor	VOCs	Low-to-Medium	BR-AT-004	22	2	1	1	26
SS054 (Building 781)	Groundwater	VOCs	High	DV-MS-0010	170	17	8	20	215

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
SS064 (Apron 2)	Groundwater	VOCs	High	DV-MS-0010	84	8 - 9	4	16	113
SS064 (Apron 2)	Surface water	VOCs	High	DV-MS-0010	48	4 - 5	2 - 3	16	72
SS068 (Building 789)	Groundwater	VOCs	High	DV-MS-0010	120	12	6	20	158
Building 785 Pipeline	Groundwater	VOCs	Low	DV-MS-0010	4	1	1	1	7
Building 785 Pipeline	Soil	VOCs	Low	DV-MS-0010	4	1	1	1	7
Building 785 Pipeline	Groundwater	SVOCs	Low	DV-MS-0012	4	1	1	1	7
Building 785 Pipeline	Soil	SVOCs	Low	DV-MS-0012	4	1	1	1	7
ST006 Building 101 AOC	SVI Vapor	VOCs	Low	BR-AT-004	40	8	4	5	52

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] The number of samples collected may vary depending on field conditions.

[3] Total numbers of Field Duplicate Samples will meet project goal of 10%.

[4] Total MS/MSD Samples will meet project goal of 5%.

[5] Trip blank samples will be included in each cooler containing aqueous VOCs.

Ambient blanks will be collected each day that VOC samples are collected.

Equipment blanks will be collected from each type of non-disposable, decontaminated sampling device.

QAPP Worksheet #21 – Project Sampling SOP References Table

SOPs are located in Appendix A.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 1	Soil Sampling	FPM/AECOM	Hand Auger or Direct Push Rig	N	Includes descriptions and procedures for surface and subsurface soil sampling.
SOP No. 2	Groundwater Sampling	FPM/AECOM	Bailer/ peristaltic pump/low flow	N	Includes descriptions and procedures for groundwater sampling.
SOP No. 3	Surface water	FPM/AECOM	Grab	N	Includes descriptions and procedures for surface water sampling.
SOP No. 4	Vapor	FPM/AECOM	Vacuum sample canister with regulator	N	Includes descriptions and procedures for SVI and soil vapor sampling.
SOP No. 5	Sample Handling, Documentation, and Tracking	FPM/AECOM	N/A	N	Includes sample packaging, shipping, and chain-of-custody requirements.
SOP No. 6	Decontamination	FPM/AECOM	N/A	N	Includes descriptions and procedures for decontamination of personnel and equipment.
SOP No. 7	Monitoring Well Installation and Development	FPM/AECOM	N/A	N	Includes description for the drilling, completion and development of monitoring wells.
SOP No. 8	Monitoring Well and Boring	FPM/AECOM	N/A	N	Includes description for the plugging and abandonment of soil borings and

QAPP Worksheet #21 – Project Sampling SOP References Table

SOPs are located in Appendix A.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
	Abandonment				monitoring wells.
SOP No. 9	Equipment Calibration	FPM/AECOM	N/A	N	Details the procedures for equipment calibration and documentation.

QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP [1]
Photoionization detector (PID)	Calibrated to 100 parts per million (ppm) using 100 ppm isobutylene	Clean unit weekly	Check response with marking pen	Observe pump and PID response	Daily	Within 3%	Clean Lamp	Field personnel	SOP No. 1, 2 and 3.
Water Quality Measure System (YSI 556 MPS, Horiba U-52, or similar)	Daily DO with tap water. Weekly conductivity with 1.413 mS/cm conductivity standard. Weekly pH with pH 4 and 7 pH standards. Weekly ORP with 240 mV ORP solution.	Clean unit daily with Simple Green or similar and distilled water rinse.	Check response to calibration.	Daily for damaged cord, probe or controller.	Daily Weekly	95-105% 1.35-1.45 mS/cm 3.98-4.02, 6.98-7.02 230 -250 mV	Clean Probe, repeat calibration procedure	Field personnel	SOP No. 3.

[1] The Project Sampling SOP References table is found on Worksheet #21.

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-IP-0010	Revision 4.7, 07/18/2012 Acid Digestion of Aqueous Samples for Metals Analysis by ICP (SW3005A)	Preparation	Metals	N/A	TA Denver	N
DV-MS-0009	Revision 3.4, 05/31/2012 Screening for VOCs by headspace GC/FID	Definitive	Volatiles	GCMS	TA Denver	N
DV-MS-0010	Revision 9.0, 01/4/2013 Determination of Volatile Organics by Gas Chromatography and Mass Spectrometer (GC/MS) (SW846 8260B and EPA 624)	Definitive	Volatiles	GCMS	TA Denver	N
DV-MS-0011	Revision 5.2, 05/04/2010 GC/MS Analysis Based on Method 8270C and 625	Definitive	Semi-Volatiles	GCMS	TA Denver	N
DV-MS-0012	Revision 3, 01/4/2013 GC/MS Analysis Based on Method 8270D	Definitive	Semi-Volatiles	GCMS	TA Denver	N
DV-WC-0006	Revision 7.1, 04/19/2010	Definitive	TOC, water	Shimadzu	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Carbon in Water (TOC, TIC, DOC, and TC) [EPA 415.1, SM 5310B & SW 9060A]					
DV-WC-0020	Revision 7.1, 12/04/2009 Anions by Ion Chromatography (EPA 300.0, SW 9056A)	Definitive	Anions	IC	TA Denver	N
DV-WC-0023	Revision 3.2, 03/01/2010 Percent Moisture in Soils and Wastes [EPA 160.3, ASTM D2216, CLP ILM05.3]	Definitive	Moisture, soils	NA	TA Denver	N
DV-WC-0082	Revision 0.2, 06/11/2010 Total and Amenable Cyanide by SW-846 9010C, 9012B, and 9013	Definitive	Cyanide	Colorimetric	TA Denver	N
DV-WC-0040	Revision 5.2, 11/19/2010 Ammonia Nitrogen by Autoanalyzer [EPA 350.1]	Definitive	Ammonia	Colorimetric	TA Denver	N
DV-WC-0060	Revision 3.2, 05/15/2010 Hardness by Titration [EPA SM2340C and SM 2340C]	Definitive	Hardness, Water	Titration	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-OP-0006	Revision 9.0, 01/15/2013 Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0007	Revision 7.0, 12/5/2012 Concentration and Clean-up of Organic Extracts (SW-846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 series)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0008	Revision 5, 08/02/2010 Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0010	Revision 3, 06/23/2010 Soxhlet Extraction of Solid Samples (SW-846 3540C)	Preparation	Organic Prep	N/A	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-OP-0015	08/1/2012 Microwave Extraction of Solid Samples (SW-846 3546)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0016	12/5/2012 Ultrasonic Extraction of Solid Samples (SW-846 3550 B and C)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0023	Revision 1, 01/31/2012 Extraction of Aqueous Samples by Microextraction, (SW-846 3511) VOCs/SVOCs	Preparation	Organic Prep	N/A	TA Denver	N
005	VOCs in Ambient Air, EPA TO-15, BR-AT-004, Rev. 7, 08/16/2012	Definitive	VOCs	GC/MS	TA Burlington	No

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
GCMS - 8260	Check of mass spectral ion intensities, i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria.	Retune the instrument and verify (instrument maintenance may be needed).	Lab Manager / Analyst	DV-MS-0010
	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for Calibration Check Compounds (CCCs) ≤ 30% and all other target analytes %RSD for RF < 15%.	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0010
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 25% of expected value	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0010
	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound; and CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression).	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.	Lab Manager / Analyst	DV-MS-0010

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). All other target compounds ≤20%, up to 5 non-CCC target compounds may fail this requirement provided the % difference is ≤ 40%.	Continuing calibration check	Lab Manager / Analyst	DV-MS-0010
	Internal Standards (IS)	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). Extracted ion current profile (EICP) area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. (See federal programs SOP DV-QA-024P for program specific requirements)	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).	Lab Manager / Analyst	DV-MS-0010
GCMS - 8270	Check of mass spectral ion intensities, i.e.,	Prior to initial calibration or Continuing	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and	Retune the instrument and verify (instrument	Lab Manager / Analyst	DV-MS-0011

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	Tune	calibration verification, every 12 hours	Pentachlorophenol requirements.	maintenance may be needed).		
	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF < 15%. option (if %RSD is > 15%)– linear regression r 2 > 0.99, r ≥ 0.995.	Correct problem then repeat initial calibration If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate.	Lab Manager / Analyst	DV-MS-0011
	ICV must be from a second source.	Immediately following five-point initial	All analytes within 25% of expected value	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0011

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
		calibration				
	CCV	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.	Lab Manager / Analyst	DV-MS-0011
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). All other target compounds ≤20%, up to 5 non-CCC target compounds may fail this requirement provided the % difference is ≤ 40%.	Continuing calibration check	Lab Manager / Analyst	DV-MS-0011
	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning	Lab Manager / Analyst	DV-MS-0011

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
			CCV for the samples. (See federal programs SOP DV-QA-024P for program specific requirements)	(dilution of the sample may be required, see the supervisor or the technical director for advice).		
Total Organic - 9060A	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	$r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0006
	ICV must be from a second source.	Immediately following initial calibration	±10%	Recalibrate	Lab Manager/ Analyst	DV-WC-0006
	CCV	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples	Lab Manager/ Analyst	DV-WC-0006
Ion Chromatograph - 9056A	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0020

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
		calibration once per year minimum.				
	ICV, second source	Immediately following initial calibration	±10%	Recalibrate	Lab Manager/ Analyst	DV-WC-0020
	CCV	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples	Lab Manager/ Analyst	DV-WC-0020
Colorimetric Analyzer - 9012B	Initial calibration (six-point calibration standards)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-WC-0082
	High and Low Distilled standard	Prepared per batch.	±10%	Re-distill and re-analyze all associated samples	Lab Manager / Analyst	DV-WC-0082
	ICV must be	Immediately	±10%	Correct problem	Lab	DV-WC-0082

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	from a second source.	following initial daily calibration		then repeat initial calibration	Manager / Analyst	
Titrimetric Analyzer - SM 2340C	Standardization of titrant	Initial daily standardization prior to sample analysis.	N/A – See method SOP for standardization procedure	N/A	Lab Manager / Analyst	DV-WC-0060
	LCS/LCSD	Prepared per batch.	±10%	Re-titrate all associated samples	Lab Manager / Analyst	DV-WC-0060
Colorimetric Analyzer - 350.1	Calibration Curve – Minimum five-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0040
	ICV must be from a second source.	Immediately following initial calibration	±10%	Recalibrate	Lab Manager/ Analyst	DV-WC-0040
	CCV	Each use, beginning, every 10 samples, end	± 10%	Rerun affected samples	Lab Manager/	DV-WC-0040

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
		of batch			Analyst	
GC/ECD HP5890 HP6890/5973	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	Initial RSD ≤20% Linear Regression r ≥ 0.995 ICV 80-120 % Recovery CCV % D or drift ≤20%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	003, 004
Leeman Labs Hydra AA	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	Linear Regression r ≥ 0.995 ICV 90-110 % Recovery CCV % D ≤20%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	001
Thermo ICAP 6500	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	ICAL NA ICV 90-110 % Recovery CCV % D ≤10%, % RSD between replicate integrations < 5%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	002
GC: Agilent 6890 MS: Agilent 5973 or 5972 MSD	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	RSD for each analyte ≤ 30% with two exceptions up to 40%	Correct problem and repeat calibration	Assigned Lab personnel	005

The Analytical SOP References table is found on Worksheet #23
1- This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2 – Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - The mean of all calibrated compounds may be used, but all compounds above the 15% must be documented in a NCM

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
GC	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	QA Manual – Section 20
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Analyst	QA Manual – Section 20
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	QA Manual – Section 20
ICP	Replace disposables,	Intensity of 1PPM	Check	Daily or as	Intensity of 1PPM	Replace, investigate	Analyst	QA Manual

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	flush lines, clean injector and torch, Perform Hg alignment, check purge windows	Manganese STD within criteria	connections	needed	Manganese STD within criteria	injector, reanalyze		– Section 20
ICP	Replace pump windings and gas tanks, check standard and sample flow	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Analyst	QA Manual – Section 20
ICPMS	Replace disposables, clean/change nebulizer, torch, and cones	Tuning	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
CVAA	Replace disposables, flush lines, check lamp current and gas flow	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
Colorimetric	Replace disposable, flush lines,	Analytical standards	Instrument performance and	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	clean autosampler and pump rollers		sensitivity					
Spectrophotometer	Replace disposable, flush lines, and clean autosampler.	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
Ion Chromatograph	Replace disposables, check for leaks and eluent levels, change columns and bed supports as needed, clean conductivity cell	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
Shimadzu	Replace disposables, check for leaks, change	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	copper and tin as needed, clean purging cell							
LECO	Replace Disposables and check gas flow.	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
HPLC	Replace columns, DAD flow cell windows and ball-valve cartridges as needed, clean/change filters, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
LC/MS & IC/MS/MS	Replace columns as needed, change filters	Sensitivity check	Instrument performance and	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	and seals, clean lenses and needles, check eluent reservoirs		sensitivity					
GC: Agilent 6890 MS: Agilent 5973 or 5972 MSD	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	TO-15	Pass Tune Pass Continuing Calibration	Daily or as needed	Tune: See TO-15 Method CCV: %D ≤ 30	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	005

QAPP Worksheet #26 – Sample Handling System

Sample Collection, Packaging, and Shipment

Sample Collection (Personnel/Organization): Field Personnel / FPM and AECOM

Sample Packaging (Personnel/Organization): Field Personnel / FPM and AECOM

Coordination of Shipment (Personnel/Organization): FPM Technical Lead / FPM and AECOM Field Personnel

Type of Shipment/Carrier: Laboratory courier/Overnight FedEx or UPS

Sample Receipt and Analysis

Sample Receipt (Personnel/Organization): TBD / Test America

Sample Custody and Storage (Personnel/Organization): TBD / Test America

Sample Preparation (Personnel/Organization): TBD / Test America

Sample Determinative Analysis (Personnel/Organization): TBD / Test America

Sample Archiving

Field Sample Storage (No. of days from sample collection): 30 days

Sample Extract/Digestate Storage (No. of days from extraction/digestion): 90 days

Biological Sample Storage (No. of days from sample collection): N/A

Sample Disposal

Personnel/Organization: TBD / Test America

Number of Days from Analysis: 30 days

QAPP Worksheet #27 – Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

See SOP No. 7.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

See the following SOPs:

SOP #LOGIN01 Sample Receiving and Login

SOP #SOP33 Laboratory Waste Management

Sample Identification Procedures:

See SOP No. 7 and Supplemental WP.

Chain of Custody Procedures:

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	VOCs and SVOCs
Analytical Method / SOP Reference	EPA 8260B/8270C/8270D DV-MS-0010, DV-MS-0011

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>1/2RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL; no common lab contaminants >RL.
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	See Table 12-2	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	EPA 9060A DV-WC-0006

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	EPA 9056A DV-WC-0020

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Duplicate	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag duplicate data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	±30%, historical or client specific limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	SM 2340C DV-WC-0060

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL; no common lab contaminants >RL.
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater
Analytical Group	Landfill Leachate Indicators
Analytical Method / SOP Reference	U.S. EPA Method SW 9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW 9060A [total organic carbon (TOC)], SM 2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW 9066 (phenols), SW 9012B (cyanide), and SW 6010B (boron)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target compounds ≥ ½ RL. For common laboratory contaminants, no analytes detected > RL.	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Laboratory Analyst	Accuracy/ Bias Contamination	No target compounds ≥ ½ RL
LCS	One per preparation/analytical batch	See Table 12-1	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst	Accuracy/ Bias	See Table 12-3
MS/MSD	Each group of field samples in an SDG or each SDG, whichever is most	See Table 12-1	Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed	Laboratory Analyst	Accuracy/Bias and Precision	See Table 12-3

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater
Analytical Group	Landfill Leachate Indicators
Analytical Method / SOP Reference	U.S. EPA Method SW 9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW 9060A [total organic carbon (TOC)], SM 2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW 9066 (phenols), SW 9012B (cyanide), and SW 6010B (boron)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
	frequent		target analytes. Examine the project-specific DQOs. Contact the client as to additional measures to be taken.			
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #28 – QC Samples Table

Matrix	Air
Analytical Group	VOC
Analytical Method / SOP Reference	TO-15/005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Blank Spike	Each batch or every 20 samples, whichever is sooner.	%R for all analytes within 70-130	Reanalyze Sample	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Method Blank	Each batch or every 20 samples.	< RL	Reanalyze Sample	TestAmerica Laboratory	Contamination	See worksheet 15 for lab LOQs
Internal Standard	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per Laboratory SOP
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #29 – Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Logbook	Sample Receipt, Custody, and Tracking Records	Sample Receipt, Custody, and Tracking Records	Field Sampling Checklists	
Boring Log	Equipment Calibration Logs	Equipment Calibration Logs	Field Analysis Audit Checklists	
COC Records	CA Forms	CA Forms	Data Validation Reports	
Air Bills	Reported Field Sample Results	Reported Field Sample Results	CA Forms	
Custody Seals	Sample Disposal Records	Reported Results for Standards, QC Checks, and QC Samples		
CA Forms	Health and Safety Inspection Forms	Data package Completeness Checklist		
Monitoring Well Construction Log		Sample Disposal Records		
		Extraction/Cleanup-up Records		
		Raw Data (stored on disk CD-R)		

QAPP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ Identification (ID) Number	Analytical SOPs	Data Package Turnaround Time	Primary Laboratory	QA Laboratory
Groundwater / Surface water	VOCs, SVOCs, and landfill leachate indicators	Low to High	LTM sites at the Former Griffiss AFB	DV-MS-0010, DV-MS-0012, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, and DV-WC-0060	20 days for full data package	TA Denver	N/A
Soil	VOCs, SVOCs	Low-to-Medium	Building 785 Pipeline	DV-MS-0010 and DV-MS-0012	20 days for full data package	TA Denver	N/A
SVI Vapor	VOCs	Low-to-Medium	SD-52 SVI System Sites and ST006	005	20 days for full data package	TA Burlington	N/A

QAPP Worksheet #31 – Planned Project Assessments Table

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Review Field Logbooks, Boring Logs, Monitoring Well Completion Logs, and COC forms	As work progresses	Internal	FPM and AECOM	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead

QAPP Worksheet #32 – Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Review Field Logbooks, Boring Logs, and Chain of Custody forms	Marked up copy of document	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	Within 24 hours of finding deficiency	Review of corrected documentation	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	24 hours after notification

QAPP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency[1] (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Activity Reports	Weekly	NA, will be included in the Annual Reports	Daniel Baldyga, FPM Technical Lead and John Santacroce, AECOM Technical Lead	
Sampling Data and Site O&M Report for Landfill AOCs	Two Annual reports. Landfill 5 and 7 in one report following the Spring Sampling Round and one for Landfill 1, 2/3, and 6 following the Fall Sampling Round.	4th Quarter of each year (calendar)	Daniel Baldyga/ FPM Technical Lead	David Farnsworth, AFCEC Project Management, Robert Morse, USEPA Remedial Project Manager, and Heather Bishop, NYSDEC Environmental Engineer
Sampling Data and Site O&M Report for On-Base Groundwater AOCs	Annually (Following Spring Sampling Round).	3rd Quarter of each year (calendar)	Daniel Baldyga/ FPM Technical Lead	
Quarterly O&M Reports for SVE and SVI Mitigation Systems	Report will be submitted following data collection and analysis.	Quarterly	Daniel Baldyga/ FPM Technical Lead	
Building 785 Pipeline Performance Monitoring Report	Report will be submitted following data collection and analysis.	3rd Quarter/2015	Daniel Baldyga/ FPM Technical Lead	David Farnsworth, AFCEC Project Management and Mark Tibbe, NYSDEC Petroleum Spills Program

QAPP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency[1] (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Sampling Data and Site O&M Report for Petroleum Spill Sites LTM and Remediation O&M	Semi-Annually (Following Spring and Fall Sampling Rounds)	Quarterly	John Santacroce/ AECOM Technical Lead	David Farnsworth, AFCEC Project Management and Mark Tibbe, NYSDEC Petroleum Spills Program

QAPP Worksheet #34 – Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
COC and shipping forms	COC forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the COC should be initialed by the reviewer, a copy of the COC retained in the project file, and the original and remaining copies taped inside the cooler for shipment. If the lab courier is used, the courier signs the COC upon receipt of sample coolers, the original is given to the lab courier and a copy is retained with the shipper.	I	Daniel Baldyga, FPM and John Santacroce, AECOM
Daily QC Reports	Upon report completion, a copy of the report will be placed in the project file.	I	Daniel Baldyga, FPM and John Santacroce, AECOM
Field Logbooks	Field logbooks will be reviewed internally and placed in the project file.	I	Daniel Baldyga, FPM and John Santacroce, AECOM
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal All received data packages will be verified externally according to the data validation procedures specified in Worksheet # 35	I E	TA

QAPP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Field Analytical Measurements	All field analytical parameters will be reviewed against the QAPP requirements for completeness and accuracy based on the field calibration records.	Daniel Baldyga, FPM and John Santacroce, AECOM
IIa	SOPs	Ensure that all sampling and analytical SOPs were followed.	Connie van Hoesel, FPM
IIb	Documentation of QC Sample Results	Establish that all required QC samples were analyzed and met evaluation criteria.	Connie van Hoesel, FPM
IIb	Project Quantitation Limits	Verify that sample results met the quantitation limits specified in the QAPP.	Connie van Hoesel, FPM

QAPP Worksheet #36 – Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
IIa	Groundwater / Surface water	VOCs, SVOCs, and landfill leachate indicators.	Low-to-High	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	Groundwater / Surface water	VOCs, SVOCs, and landfill leachate indicators.	Low-to-High	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1 through 12-6	Connie van Hoesel, FPM
IIa	Soil	VOCs and SVOCs	Low-to-Medium	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	Soil	VOCs and SVOCs	Low-to-Medium	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1 through 12-6	Connie van Hoesel, FPM
IIa	SVI Vapor	VOCs	Low-to-Medium	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	SVI Vapor	VOCs	Low-to-Medium	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1, 12-2 and 12-7	Connie van Hoesel, FPM

QAPP Worksheet #37 – Usability Assessment

A complete (100%) data review will be performed on the samples collected during the sampling event. The review will consist of verification and validation based on completeness and compliance checks of sample receipt conditions and both sample-related and instrument-related QC results, as addressed in Worksheet 12. The Data Usability Assessment will be performed by FPM personnel. Connie van Hoesel, FPM Chemical QC Coordinator will be responsible for information in the Usability Assessment. Note that the Data Usability Assessment will be conducted on verified/validated data. After the Data Usability Assessment has been performed, data deemed appropriate for decision-making purposes will be used to assess contaminant extents at sites at the former Griffiss AFB. The results of the Data Usability Assessment will be presented in the Site Specific LTM Report. The following items will be assessed and conclusions drawn based on their results.

Precision: Results of field duplicates will be presented separately in tabular format for each sample pair. For each field duplicate pair, the results will be assessed as stated in Tables 12-3 through 12-5. MS/MSD RPDs are calculated by the laboratory and those with RPDs outside the criteria established in Table 12-1 will be listed in tabular form in the data verification report. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described.

Accuracy/Bias Contamination: Results for all laboratory method blanks will be evaluated and analytes detected in these blanks will be listed in tabular form in the data verification report. Laboratory data will be qualified based on the criteria listed in Tables 12-3 through 12-5. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias: Results for all laboratory control sample (LCS), surrogate and MS/MSD recoveries that are outside evaluation criteria will be presented in tabular format in the data verification reports. The results will be checked versus those listed in Tables 12-1 and 12-2. A discussion will follow summarizing the overall accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Representativeness: A measure of representativeness will be provided by assessing if the proper analytical procedures, appropriate methods, laboratory SOPs, holding times and field duplicates are followed. Any conclusions about the representativeness of the analyses will be drawn and any limitations on the use of the data will be described.

Comparability: In accordance with this UFP QAPP the data are comparable when collection techniques, measurement method and reporting

QAPP Worksheet #37 – Usability Assessment

procedures are the same for each data set.

Completeness: A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated as the number of data points for each analyte that is deemed useable (not rejected) divided by the total number of data points for each analyte. A discussion will follow summarizing the results of the calculation of data completeness. Any conclusions about the completeness of the data will be drawn and any limitations on the use of the data will be described. Data completeness addresses only those samples that are collected and only data that is analyzed by the laboratory.

Graphics: Figures and maps will be prepared showing the fuel oil impact levels at each sampling location.

Reconciliation: Each of the measurement performance criteria listed in Worksheet #12 will be examined to determine if the objective was met. Each analysis will be evaluated separately in terms of the major impacts observed from the data verification/validation, data quality indicators and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Usability of the data will be based on the quality assessment. After establishing the usability of the data, it will be determined if the DQO was met and if project action limits were met. The final report will include a summary of all points that comprised the reconciliation of each objective. Any conclusions or limitations on the usability of any of the data will be described.

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Tables

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
VOCs				
1,1,1,2-Tetrachloroethane	80 - 130	75 - 125	30	30
1,1,1-Trichloroethane	65 - 130	70 - 135	30	30
1,1,2,2-Tetrachloroethane	65 - 130	55 - 130	30	30
1,1,2-Trichloro-1,2,2-trifluoroethane	N/A	N/A	30	30
1,1,2-Trichloroethane	75 - 125	60 - 125	30	30
1,1-Dichloroethane	70 - 135	75 - 125	30	30
1,1-Dichloroethene	70 - 130	65 - 135	30	30
1,1-Dichloropropene	75 - 130	70 - 135	30	30
1,2,3-Trichlorobenzene	55 - 140	60 - 135	30	30
1,2,3-Trichloropropane	75 - 125	65 - 130	30	30
1,2,4-Trichlorobenzene	65 - 135	65 - 130	30	30
1,2,4-Trimethylbenzene	75 - 130	65 - 135	30	30
1,2-Dibromo-3-chloropropane	50 - 130	40 - 135	30	30
1,2-Dibromoethane (EDB)	80 - 120	70 - 125	30	30
1,2-Dichlorobenzene	70 - 120	75 - 120	30	30
1,2-Dichloroethane	70 - 130	70 - 135	30	30
1,2-Dichloropropane	75 - 125	70 - 120	30	30
1,3,5 -Trimethylbenzene	75 - 130	65 - 135	30	30
1,3-Dichlorobenzene	75 - 125	70 - 125	30	30
1,3-Dichloropropane	75 - 125	75 - 125	30	30
1,4-Dichlorobenzene	75 - 125	70 - 125	30	30
1,4-Dioxane	N/A	N/A	30	30
1-Chlorohexane	N/A	N/A	30	30
2,2-Dichloropropane	70 - 135	65 - 135	30	30
2-Butanone	30 - 150	30 - 160	30	30
2-Chlorotoluene	75 - 125	70 - 130	30	30
2-Hexanone	55 - 130	45 - 145	30	30
4-Chlorotoluene	75 - 130	75 - 125	30	30
4-Methyl-2-pentanone	60 - 135	45 - 145	30	30
Acetone	40 - 140	20 - 160	30	30
Benzene	80 - 120	75 - 125	30	30
Bromobenzene	75 - 125	65 - 120	30	30
Bromochloromethane	65 - 130	70 - 125	30	30
Bromodichloromethane	75 - 120	70 - 130	30	30
Bromoform	70 - 130	55 - 135	30	30
Bromomethane	30 - 145	30 - 160	30	30
Carbon disulfide	35 - 160	45 - 160	30	30
Carbon tetrachloride	65 - 140	65 - 135	30	30
Chlorobenzene	80 - 120	75 - 125	30	30
Chloroethane	60 - 135	40 - 155	30	30
Chloroform	65 - 135	70 - 125	30	30
Chloromethane	40 - 125	50 - 130	30	30
cis-1,2-Dichloroethene	70 - 125	65 - 125	30	30
cis-1,3-Dichloropropene	70 - 130	70 - 125	30	30
Cyclohexane	N/A	N/A	30	30
Dibromochloromethane	60 - 135	65 - 130	30	30
Dibromomethane	75 - 125	75 - 130	30	30
Dichlorodifluoromethane	30 - 155	35 - 135	30	30
Ethylbenzene	75 - 125	75 - 125	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
Hexachlorobutadiene	50 - 140	55 - 140	30	30
Isopropylbenzene	75 - 125	75 - 130	30	30
Methyl acetate	N/A	N/A	30	30
Methylene Chloride	55 - 140	55 - 140	30	30
Methyl-tert-butyl Ether	65 - 125	67 - 119	30	30
Methylcyclohexane	N/A	N/A	30	30
m-p-Xylene	75 - 130	80 - 125	30	30
Naphthalene	55 - 140	40 - 125	30	30
n-Butylbenzene	70 - 135	65 - 140	30	30
n-Propylbenzene	70 - 130	65 - 135	30	30
o-Xylene	80 - 120	75 - 125	30	30
p-Isopropyltoluene	75 - 130	75 - 135	30	30
sec-Butylbenzene	70 - 125	65 - 130	30	30
Styrene	65 - 135	75 - 125	30	30
tert-Butylbenzene	70 - 130	65 - 130	30	30
Tetrachloroethene	45 - 150	65 - 140	30	30
Toluene	75 - 120	70 - 125	30	30
trans-1,2-Dichloroethene	60 - 140	65 - 135	30	30
trans-1,3-Dichloropropene	55 - 140	65 - 125	30	30
Trichloroethene	70 - 125	75 - 125	30	30
Trichlorofluoromethane	60 - 145	25 - 185	30	30
Vinyl chloride	50 - 145	60 - 125	30	30
Xylenes (total)	80 - 120	70 - 130	30	30
SVOCs/PAHs				
1,1'-Biphenyl	N/A	N/A	30	30
1,2,4,5-Tetrachlorobenzene	N/A	N/A	30	30
1,2,4-Trichlorobenzene	35 - 105	45 - 110	30	30
1,2-Dichlorobenzene	35 - 100	45 - 100	30	30
1,3-Dichlorobenzene	30 - 100	40 - 100	30	30
1,4-Dichlorobenzene	30 - 100	35 - 105	30	30
2,3,4,6-Tetrachlorophenol	N/A	N/A	30	30
2,4,5-Trichlorophenol	50 - 110	50 - 110	30	30
2,4,6-Trichlorophenol	50 - 115	45 - 110	30	30
2,4-Dichlorophenol	50 - 105	45 - 110	30	30
2,4-Dimethylphenol	30 - 110	30 - 105	30	30
2,4-Dinitrophenol	15 - 140	15 - 130	30	30
2,4-Dinitrotoluene	50 - 120	50 - 115	30	30
2,6-Dinitrotoluene	50 - 115	50 - 110	30	30
2-Chloronaphthalene	50 - 105	45 - 105	30	30
2-Chlorophenol	35 - 105	45 - 105	30	30
2-Methylnaphthalene	45 - 105	45 - 105	30	30
2-Methylphenol	40 - 110	40 - 105	30	30
2-Nitroaniline	50 - 115	45 - 120	30	30
2-Nitrophenol	40 - 115	40 - 110	30	30
3,3'-Dichlorobenzidine	20 - 110	10 - 130	30	30
3/4-Methylphenol	30 - 110	40 - 105	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
3-Nitroaniline	20 - 125	25 - 110	30	30
4,6-Dinitro-2-methylphenol	40 - 130	30 - 135	30	30
4-Bromophenyl phenyl ether	50 - 115	45 - 115	30	30
4-Chloro-3-methylphenol	45 - 110	45 - 115	30	30
2/4-Chloroaniline	15 - 110	10 - 95	30	30
4-Chlorophenyl phenyl ether	50 - 110	45 - 110	30	30
4-Nitroaniline	35 - 120	35 - 115	30	30
4-Nitrophenol	10 - 125	15 - 140	30	30
Acenaphthylene	50 - 105	45 - 105	30	30
Acenapthene	45 - 110	45 - 110	30	30
Acetophenone	N/A	N/A	30	30
Anthracene	55 - 110	55 - 105	30	30
Atrazine	N/A	N/A	30	30
Benzaldehyde	N/A	N/A	30	30
Benzo(a)anthracene	55 - 110	50 - 110	30	30
Benzo(a)pyrene	55 - 110	50 - 110	30	30
Benzo(b)fluoranthene	45 - 120	45 - 115	30	30
Benzo(g,h,i)perylene	40 - 125	40 - 125	30	30
Benzo(k)fluoranthene	45 - 125	45 - 125	30	30
Benzoic acid	10 - 125	10 - 110	30	30
Benzyl alcohol	30 - 110	20 - 125	30	30
bis(2-Chloroethoxy) methane	45 - 105	45 - 110	30	30
bis(2-Chloroethyl) ether	35 - 110	40 - 105	30	30
bis(2-Chloroisopropyl) ether (2,2'-oxybi	25 - 130	20 - 115	30	30
bis(2-Ethylhexyl) phthalate	40 - 125	45 - 125	30	30
Butyl benzyl phthalate	45 - 115	50 - 125	30	30
Caprolactam	N/A	N/A	30	30
Carbazole	50 - 115	45 - 115	30	30
Chrysene	55 - 110	55 - 110	30	30
Dibenz(a,h)anthracene	40 - 125	40 - 125	30	30
Dibenzofuran	55 - 105	50 - 105	30	30
Diethyl phthalate	40 - 120	50 - 115	30	30
Dimethyl phthalate	25 - 125	50 - 110	30	30
Di-n-butyl phthalate	55 - 115	55 - 110	30	30
Di-n-octyl phthalate	35 - 135	40 - 130	30	30
Fluoranthene	55 - 115	55 - 115	30	30
Fluorene	50 - 110	50 - 110	30	30
Hexachlorobenzene	50 - 110	45 - 120	30	30
Hexachlorobutadiene	25 - 105	40 - 115	30	30
Hexachlorocyclopentadiene	10 - 82	35 - 123	30	30
Hexachloroethane	30 - 100	35 - 110	30	30
Indeno(1,2,3-cd)pyrene	45 - 125	40 - 120	30	30
Isophorone	50 - 110	45 - 110	30	30
Naphthalene	40 - 100	40 - 105	30	30
Nitrobenzene	45 - 110	40 - 115	30	30
N-Nitrosodi-n-propylamine	35 - 130	40 - 115	30	30
N-Nitrosodiphenylamine	50 - 110	50 - 115	30	30
Pentachlorophenol	40 - 115	25 - 120	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
Phenanthrene	50 - 115	50 - 110	30	30
Phenol	10 - 115	40 - 100	30	30
Pyrene	50 - 130	45 - 125	30	30
Inorganics				
Bromide	86 - 110		10	
Chloride	89 - 110		10	
Nitrate	87 - 110		10	
Sulfate	86 - 110		10	
Cyanide	90 - 110		10	
TOC	86 - 114		12	
TKN	77 - 115		25	
BOD	85 - 115		20	
COD	90 - 110		11	
Alkalinity	90 - 110		10	
TDS	86 - 110		20	
Hardness	90 - 110		10	
Soil Gas VOCs	%R (gas)		RPD (gas)	
1,1,1-TCA	70-130		25	
1,2-DCA	70-130		25	
1,2-Dibromoethane	70-130		25	
Benzene	70-130		25	
Carbon tetrachloride	70-130		25	
Chloroform	70-130		25	
Styrene	70-130		25	
TCE	70-130		25	
m,p-Xylene	70-130		25	
o-Xylene	70-130		25	
Tetrachloroethylene	70-130		25	
Toluene	70-130		25	
Ethylbenzene	70-130		25	
cis-1,2-DCE	70-130		25	
Methylene chloride	70-130		25	
Chloromethane	70-130		25	
Chloroethane	70-130		25	
Vinyl Chloride	70-130		25	
1,1,2,2-Tetrachloroethane	70-130		25	
1,1-Dichloroethene	70-130		25	
1,1,2-Trichloroethane	70-130		25	
1,1-DCA	70-130		25	
1,2-Dichloropropane	70-130		25	
trans-1,2-DCE	70-130		25	

\

Control limits based on DOD QSM, when available; otherwise, based on the laboratory's in-house limits.

%R - Percent Recovery

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil

N/A - Not Applicable

PAHs - Polynuclear Aromatic Hydrocarbons

PCBs - Polychlorinated Biphenyls

RPD - Relative Percent Difference

SVOCs - Semi-Volatile Organic Compounds

VOCs - Volatile Organic Compounds

TABLE 12-2
ORGANIC SURROGATE COMPOUND ACCURACY CRITERIA
FORMER GRIFFISS AIR FORCE BASE

Analysis	Spiking Compound	Accuracy (%R)	
		Aqueous	Soil
VOCs	1,2-Dichloroethane-d_4	70 - 120	80 - 120
	4-Bromofluorobenzene	75 - 120	85 - 120
	Toluene-d_8	85 - 120	85 - 115
	Dibromofluoromethane	85 - 115	80 - 120
SVOCs/PAHs	2,4,6-Tribromophenol	40 - 125	35 - 125
	2-Fluorobiphenyl	50 - 110	45 - 105
	2-Fluorophenol	20 - 110	35 - 105
	Nitrobenzene-d_5	40 - 110	35 - 100
	Phenol-d_5	10 - 115	40 - 100
	Terphenyl-d_{14}	50 - 135	30 - 125

Control limits based on DOD QSM, when available; otherwise, based on the laboratory's in-house limits.

%R - Percent Recovery
N/A - Not Applicable
PAHs - Polynuclear Aromatic Hydrocarbons
PCBs - Polychlorinated Biphenyls
SVOCs - Semi-Volatile Organic Compounds
VOCs - Volatile Organic Compounds

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	Soil VOCs: 7 days unpreserved, 14 days preserved. Soil SVOCs: 14 days to extract, 40 days to analysis Water VOCs: 7 days unpreserved, 14 days preserved. Water SVOCs: 7 days to extract, 40 days to analysis.	Contact FPM as to additional measures to be taken.		Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Sample temperature	Every cooler	4±2 °C	Contact FPM as to additional measures to be taken.	None	Samples arriving at temperature 6-10°C, apply **J**-flag to detects and **UJ**-flag to nondetects. Samples arriving at temperature > 10°C, apply **J**-flag to detects and **R**-flag to nondetects (SVOCs only). VOC samples received at temperature > 10°C, **R**-flag all results.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Problem must be corrected. No samples may be accepted without a valid tune.	Apply **R-**flag to data without a valid tune
Breakdown check (DDT Method 8270C only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check	No samples shall be run until degradation ≤ 20%.	Apply **R-**flag to data without a valid breakdown check

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Minimum five point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	1. Average response factor (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.	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed	Apply **R-**flag to data without a valid ICAL
		2. RSD for RFs for CCCs: VOCs and SVOCs - ≤ 30% and one option below; Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression - coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order)			Apply **R-**flag to data without a valid ICAL
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flag to data without second source verification.

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Evaluation of relative retention times	Each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance.	Apply **R**-flag to data outside retention time window
Manual Integration	All	Acceptance by FPM Chemist	Provide justification for each instance of manual integration	Laboratory will provide chromatograms before and after each manual integration	Apply **R**-flag to all compounds with improper integration
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time.	Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050.			Apply **J**-flag to detects and **UJ**-flag to nondetects if average RF not met
		% Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D ≤ difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Reanalyze all samples since last acceptable CCV.	Problem must be corrected. No results may be reported without a valid CCV. Flagging criteria is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **R**-flag to nondetects
Internal standards verification	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the CV	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Sample results are not acceptable without a valid IS verification.	If corrective action fails in field samples, apply **J**-flag to detects and **UJ**-flag to nondetects to analytes with IS recoveries between 30%-50% or > 150%. Apply **R**-flag to samples with IS recoveries < 30%.
		EICP area within - 50% to + 100% of ICAL midpoint standard			

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination (<10X for common laboratory contaminants).
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact URS as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD $\leq$ 30% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.	Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Surrogate spike	All field and QC samples	QC acceptance criteria specified in UFP-QAPP Table 12-2.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	Analytes identified in UFP-QAPP Table 12-2.	High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<10%): Apply **J**-flag to detects and **R**-flag to nondetects.
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2).	N/A	N/A	Appy **J**-flag to detects and **UJ**-flag to nondetects.

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	48 hours (nitrate/nitrite)/28 days (chloride, bromide, sulfate); see Worksheet #19	Contact FPM as to additional measures to be taken.		Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Initial calibration for all analytes (ICAL) Minimum three standards and one calibration blank	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed.	Apply **R-**flag to data without a valid ICAL
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analytes within ± 10% of expected value (initial source) and retention times within appropriate windows	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flag to data without second source verification

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Midrange Continuing Calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence.	All analytes within ± 10% of expected value from ICAL. All project analytes within established retention time windows.	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **R**-flag to data with CCV outside criteria.
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination.
Laboratory control sample (LCS)	One per preparatory batch	Laboratory in-house limits not to exceed ±20%. Control limits may be not greater than ±3 x the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1 (not to exceed ±20%).	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. No data flagging if native concentrations are > 4X spiking	For the specific analyte(s) in the batch. High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD ≤ 15% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	Data shall be evaluated to determine the source of difference.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Sample Duplicate	One per 10 field samples	RPD $\leq$ 10% (sample and sample duplicate)	Correct problem and reanalyze sample and duplicate.	N/A	Apply **J**-flag to detects and **UJ**-flag to nondetects if sample cannot be rerun or reanalysis does not correct problem.

TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	30 days	Contact FPM as to additional measures to be taken.	None None	Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
MS tuning check (Use BFB)	Prior to initial calibration and calibration verification	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Problem must be corrected. No samples may be accepted without a valid tune.	Apply **R-**flag to data without a valid tune
Minimum five point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	RSD for each analyte $\leq$ 30% with at most 2 exceptions up to 40%	Correct problem then repeat initial calibration	None	Apply **R-**flag to data without a valid ICAL
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 30% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flags to data without second source verification.

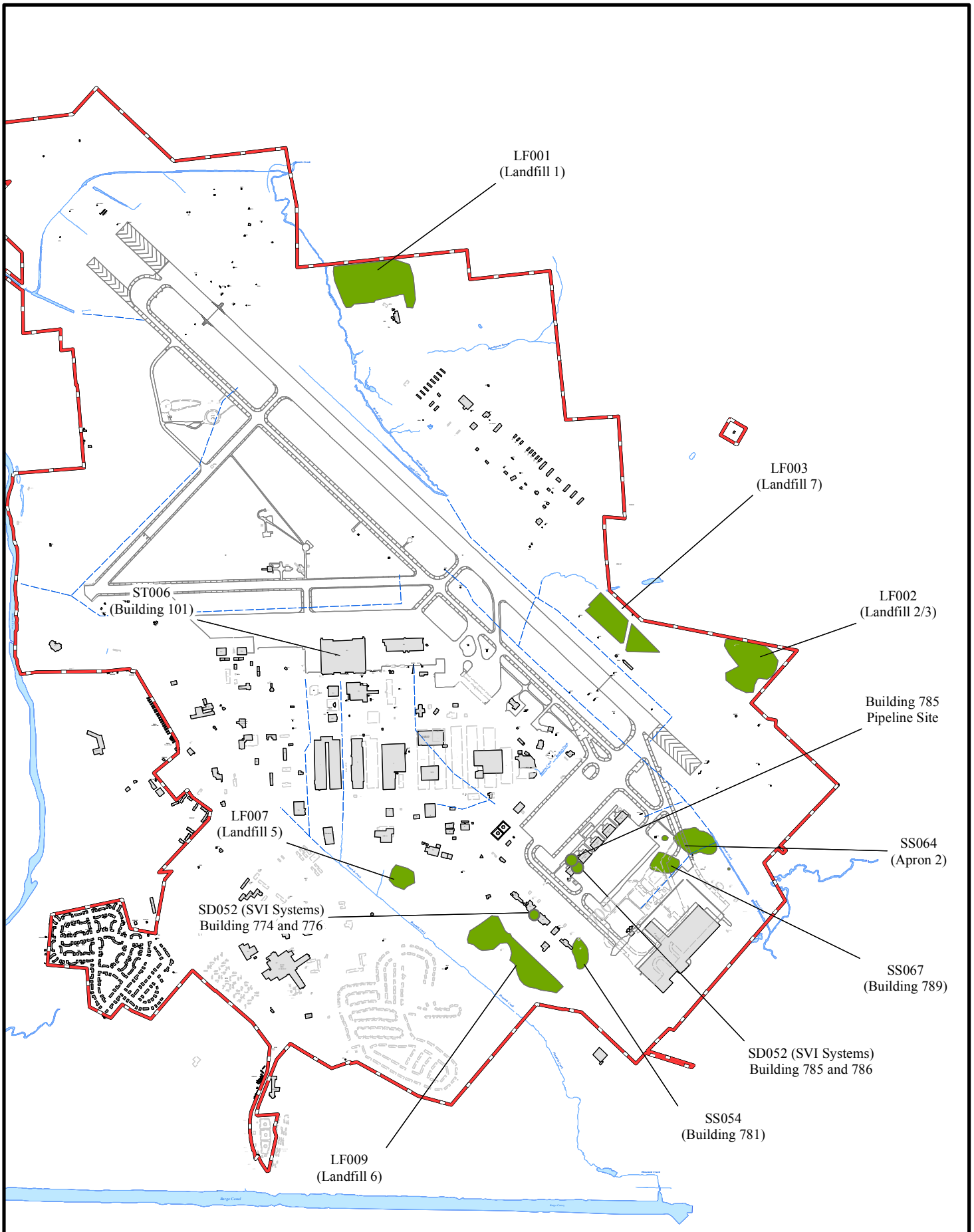
TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Manual Integration	All	Acceptance by FPM Chemist	Provide justification for each instance of manual integration	Laboratory will provide chromatograms before and after each manual integration	Apply **R**-flag to all compounds with improper integration
Calibration verification (CCV)	Prior to sample analysis (unless ICAL performed on same day), and every 24 hours of analysis time	All analytes within ± 30% of expected value form ICAL	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **R**-flag to nondetects.
Method blank (humid zero air)	Immediately after ICAL or daily CCV	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination.
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	LCS not required per method. The laboratory performs an LCS as an evaluation of percent recovery in a blank matrix in approximately 1 of every 20 samples.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.

TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD $\leq$ 25% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.	Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.
Internal standards (ISs)	Each sample	Retention time ±0.33 minutes from retention time of the IS in the most recent valid calibration. (ICAL mid-point standard or CCV) EICP area within ±40% of area of the IS in most recent valid calibration	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Sample results are not acceptable without a valid IS verification.	If corrective action fails in field samples, apply J-flag to detects and UJ-flag to nondetects to analytes with IS recoveries between 30%-60% or > 140%. Apply R-flag to samples with IS recoveries < 30%.
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)	N/A	N/A	Appy **J**-flag to detects and **UJ**-flag to nondetects.

Figures


LF001
(Landfill 1)
LF003
(Landfill 7)
LF002
(Landfill 2/3)
ST006
(Building 101)
Building 785
Pipeline Site
LF007
(Landfill 5)
SS064
(Apron 2)
SD052 (SVI Systems)
Building 774 and 776
SS067
(Building 789)
SD052 (SVI Systems)
Building 785 and 786
SS054
(Building 781)
LF009
(Landfill 6)
Barge Canal
Oneida County
Base Location
N
Map is projected in accordance with the State Plane New York Central - 3102 (NAD 83, Feet)
Key to Features
Culvert / Ditch
Surface Water
Base Boundary
Demolished Facility
Existing Facility
Griffiss Sites
0 500 1,000 2,000 3,000
Feet
UNITED STATES AIR FORCE
FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK
Figure 17-1
Site Location Map
March 2015
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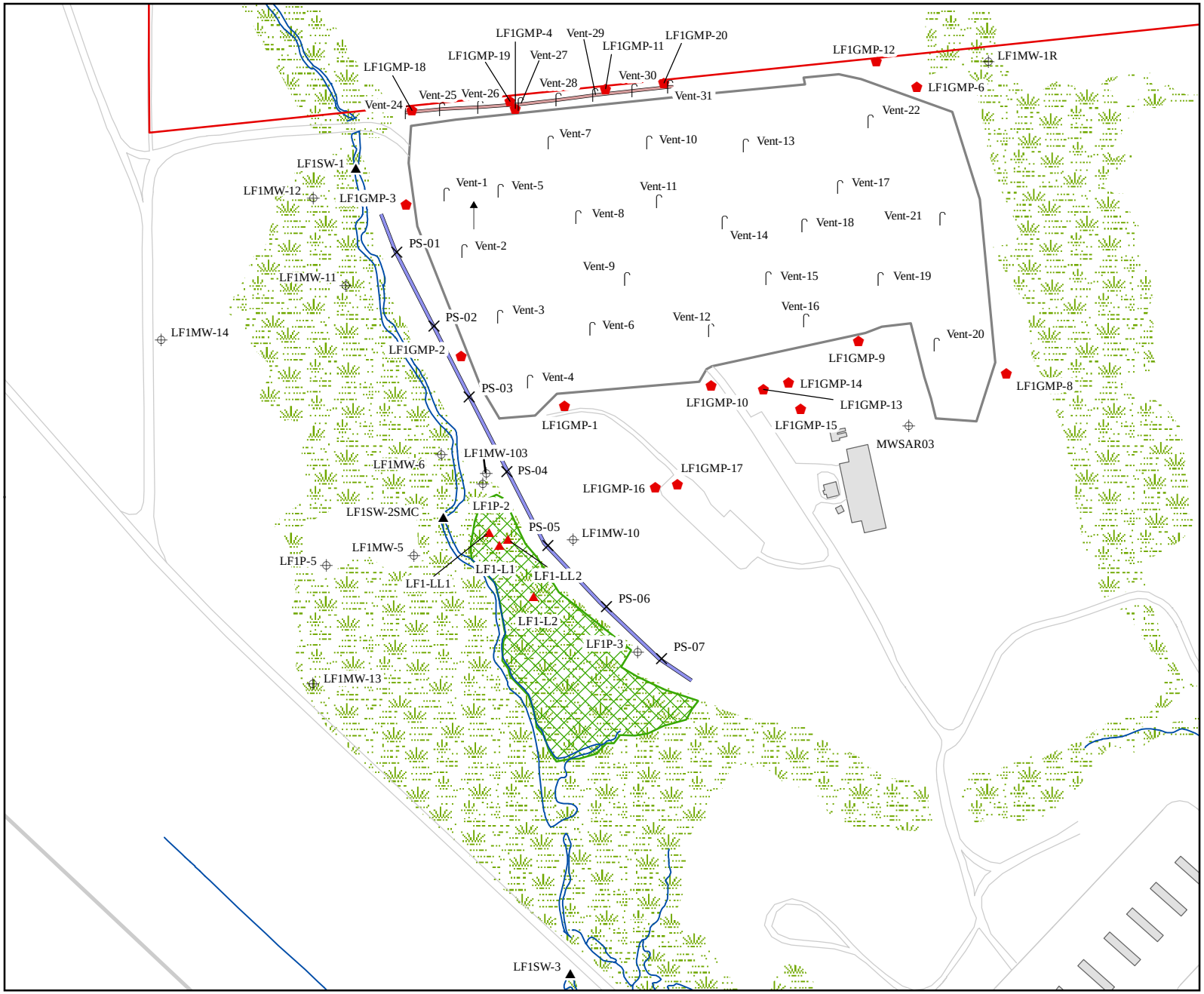


N

Site Location

Legend

- Pump Station and Sampling Location
- Leachate Sampling Location
- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- Gas Monitoring Probe with ID
- Gas Vent with ID
- Leachate Collection Trench
- Passive Gas Collection Trench
- AFB Boundary
- Surface Water
- Road
- Airfield
- Landfill Boundary
- Building
- Rich Sloping Fen
- Wetland Area

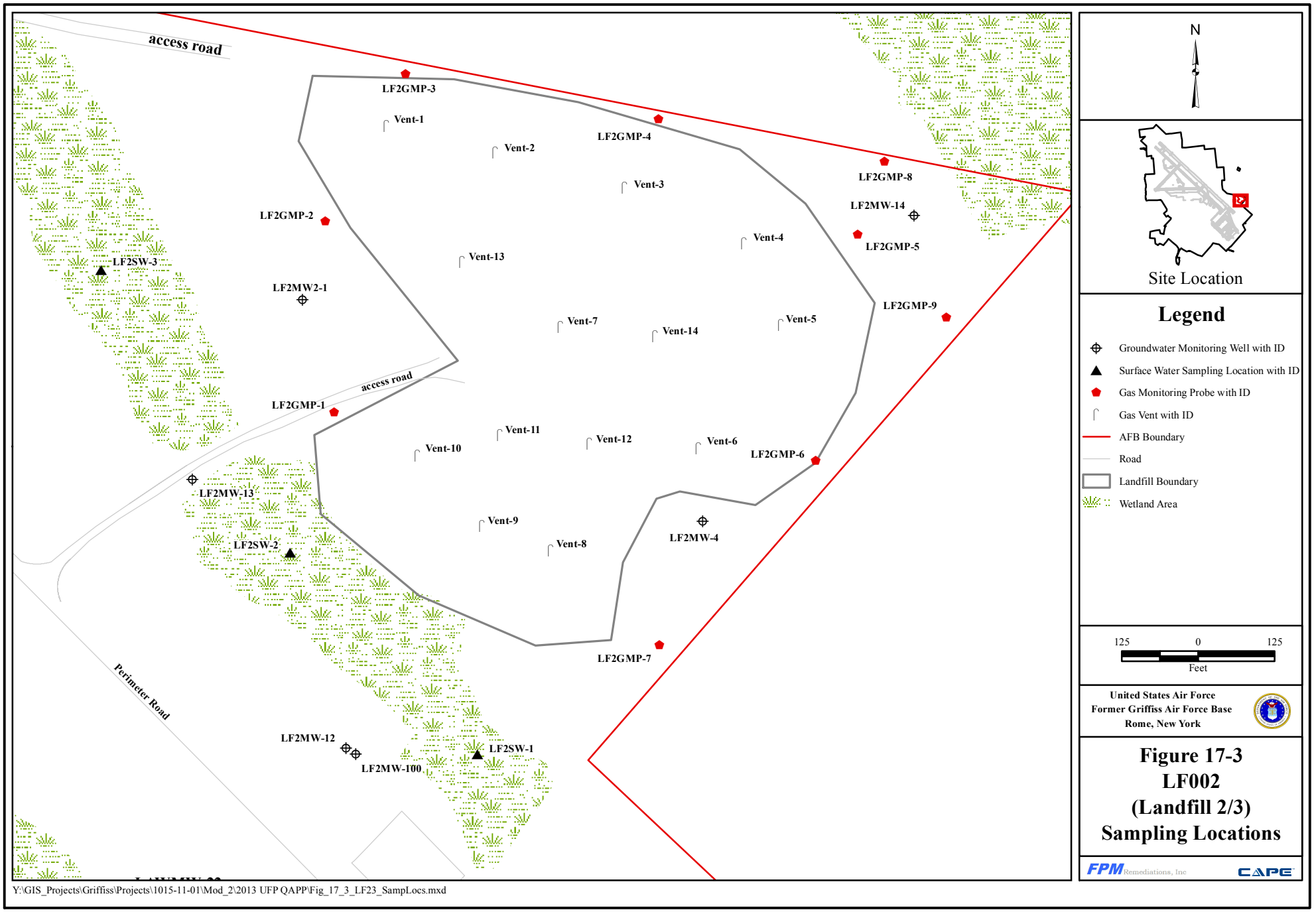
250 0 250
Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-2
LF001
(Landfill 1)
Sampling Locations

FPM Remediation, Inc. CAPE

Y:\GIS_Projects\Griffiss\Projects\1015-11-01\Mod_2\2013 UFP QAPP\Fig_17_2_LF1_SampLocs.mxd



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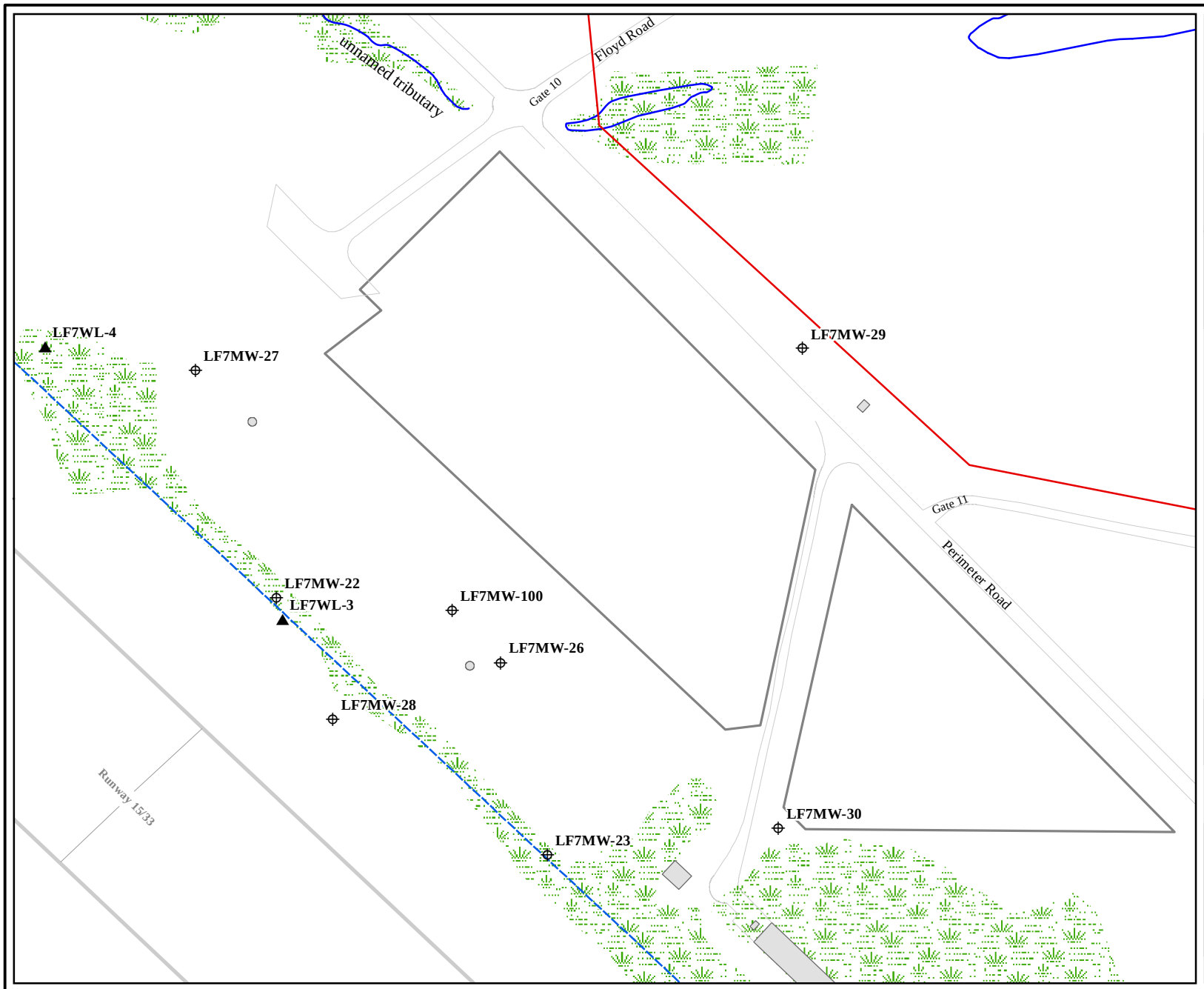

N

Site Location

Legend

- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- AFB Boundary
- Surface Water
- Storm Drain
- Road
- Airfield
- Landfill Boundary
- Building
- Wetland

150 0 150

Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-4
LF003
(Landfill 7)
Sampling Locations

FPM CAPE

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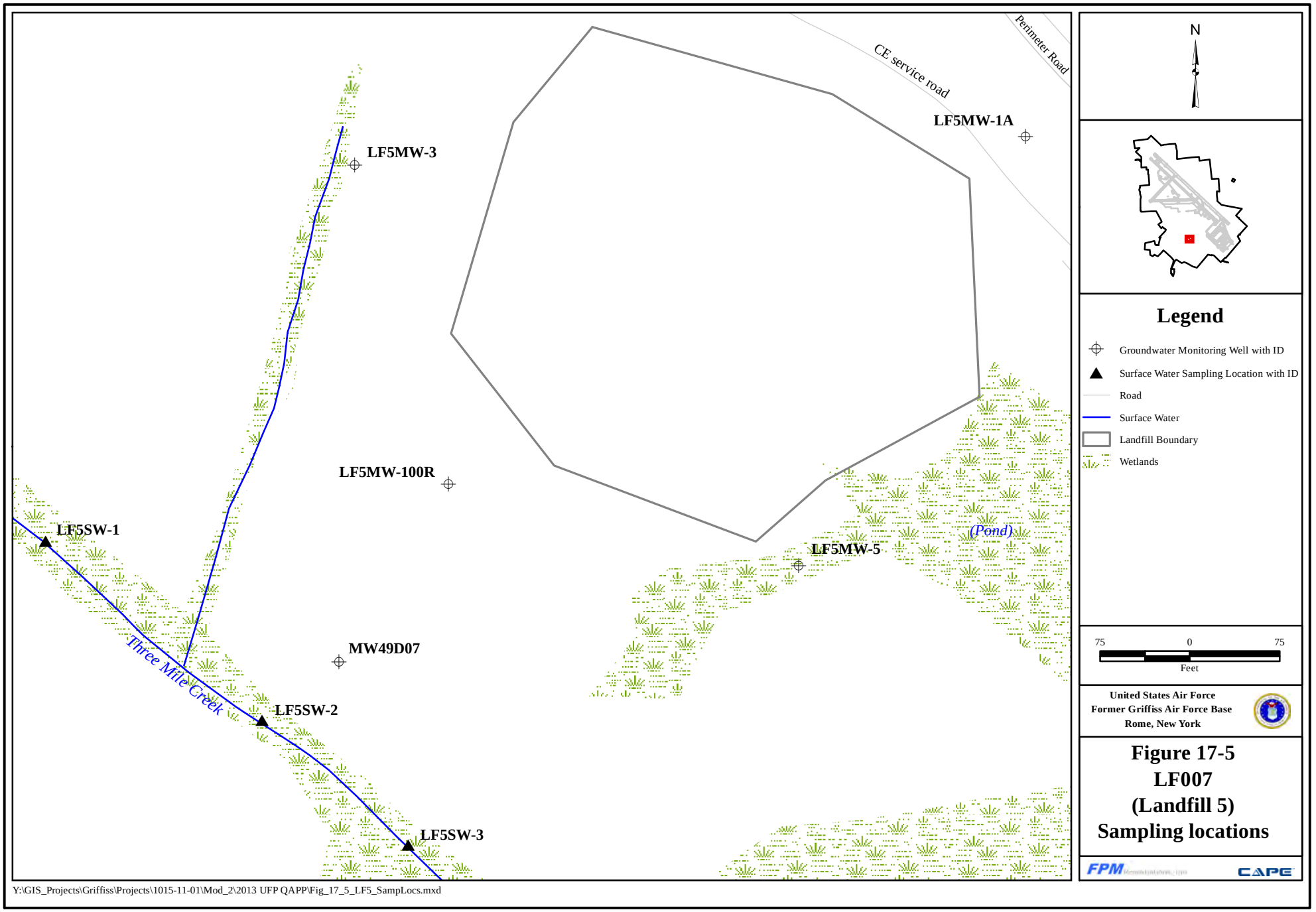

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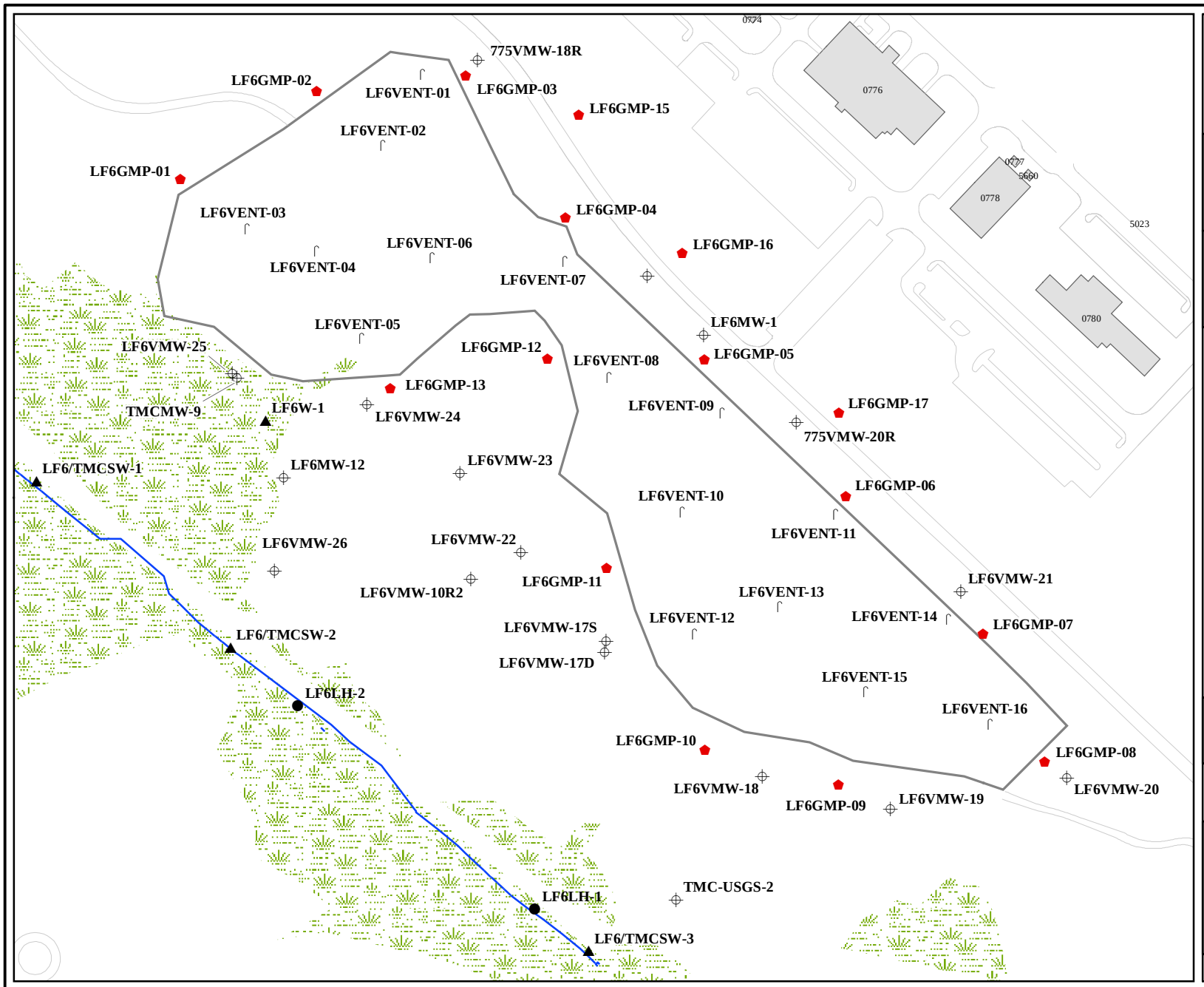

N

Legend

- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- Leachate Sampling Location with ID
- Gas Monitoring Probe with ID
- Gas Vent with ID
- Surface Water
- Road
- Landfill Boundary
- Building
- Wetland Area

200 0 200
Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-6
LF009
(Landfill 6)
Sampling Locations

FPM CAPE

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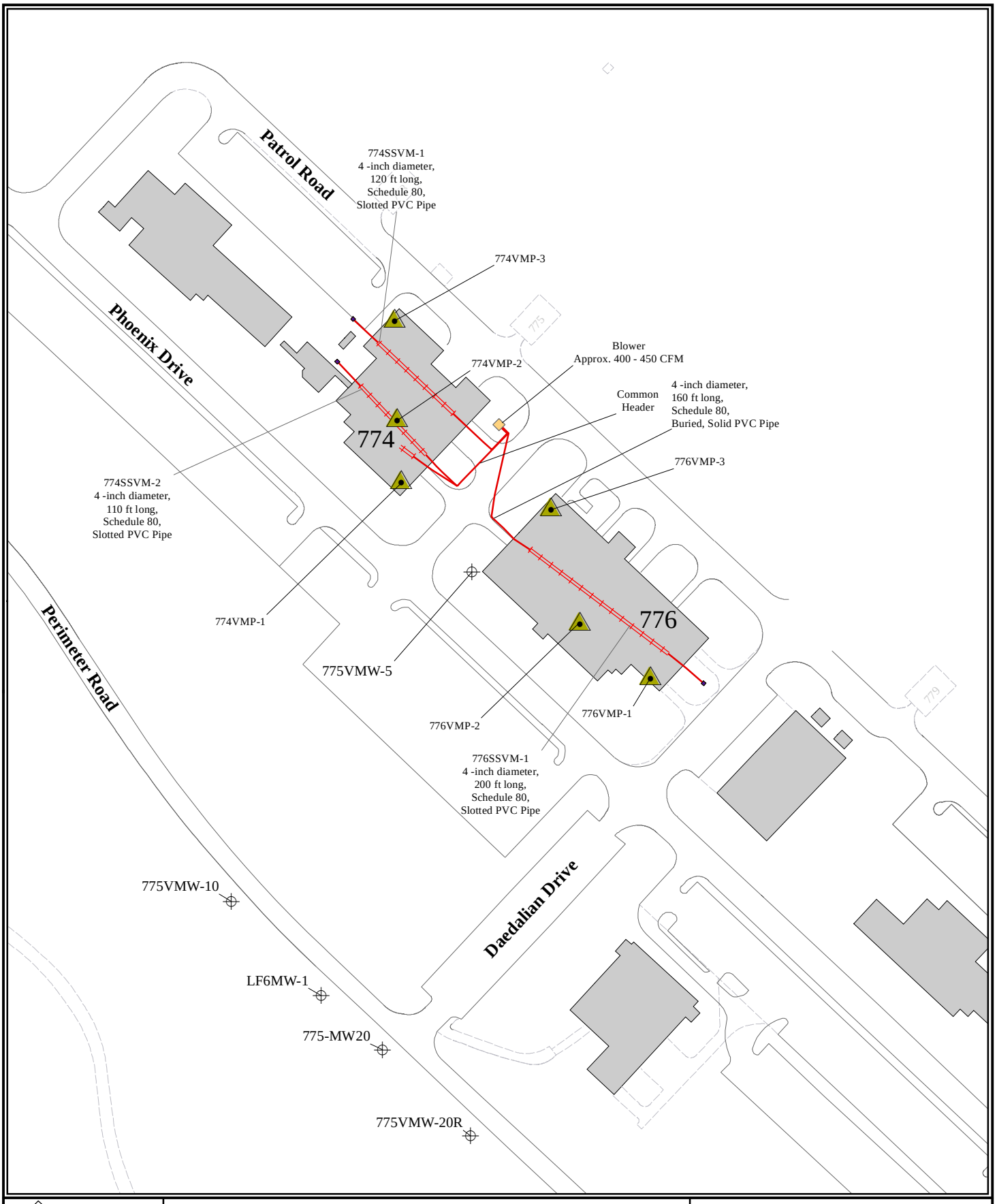

Site Location

Key to Features

- 2011 Sample Location
- Monitoring Well
- Demolished Road
- Existing Road
- Horizontal Well
- Screen
- Riser
- SVE System
- Entrance/Exit Pit
- Demolished Facility
- Existing Facility

N

100 50 0 100 Feet

UNITED STATES AIR FORCE
GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-7
SD052
(SVI Systems)
Building 774 and 776
Sampling Locations

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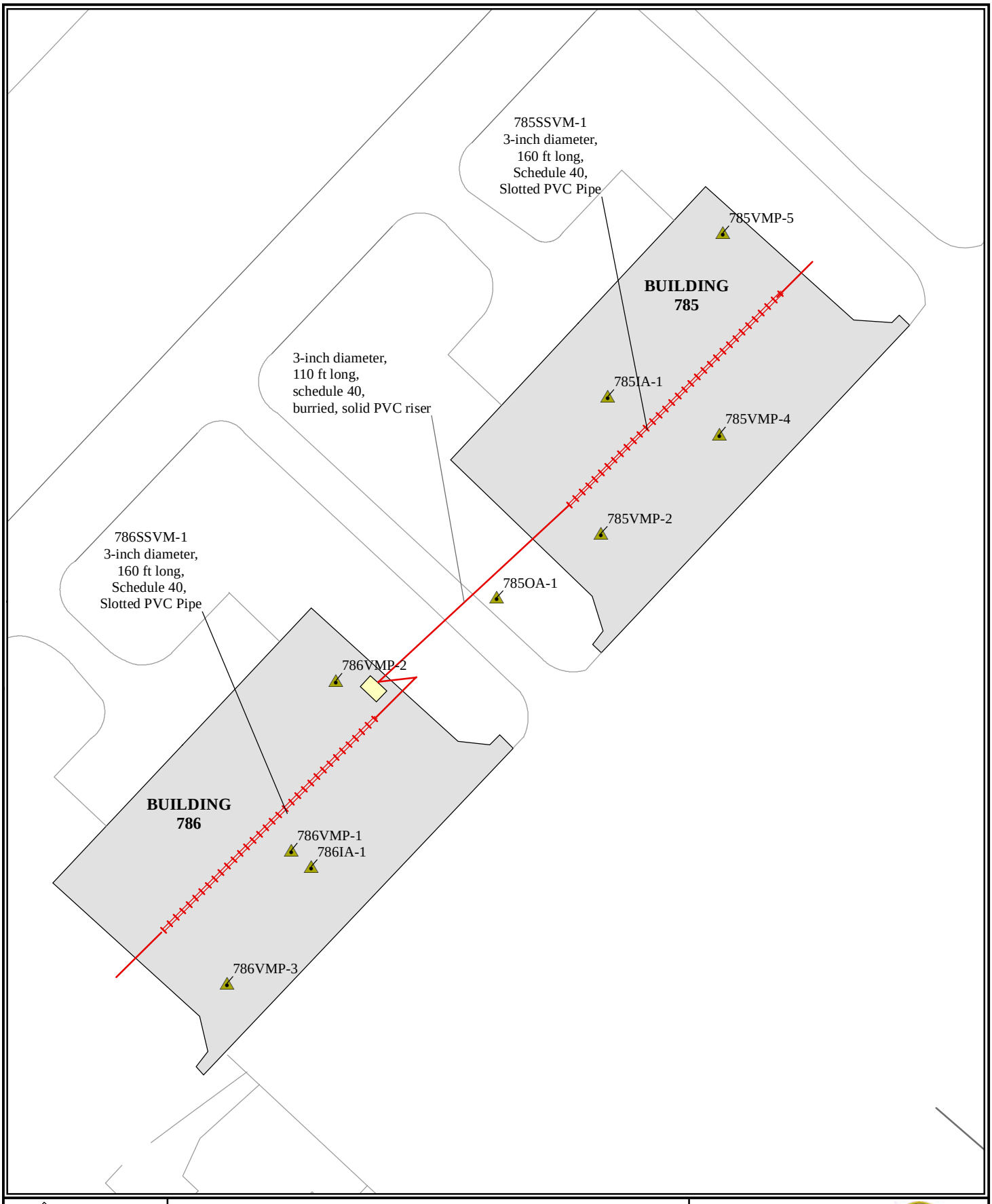

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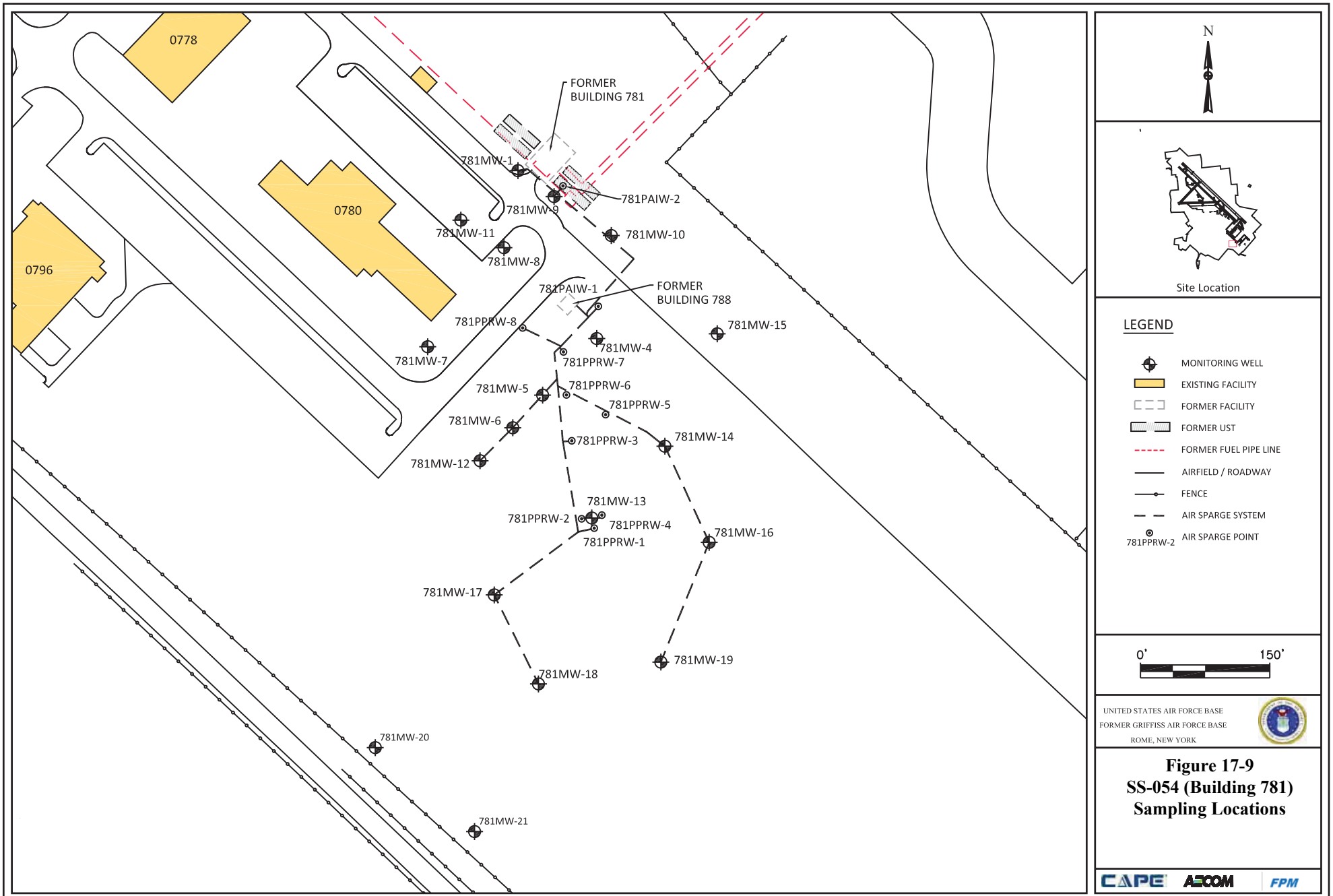

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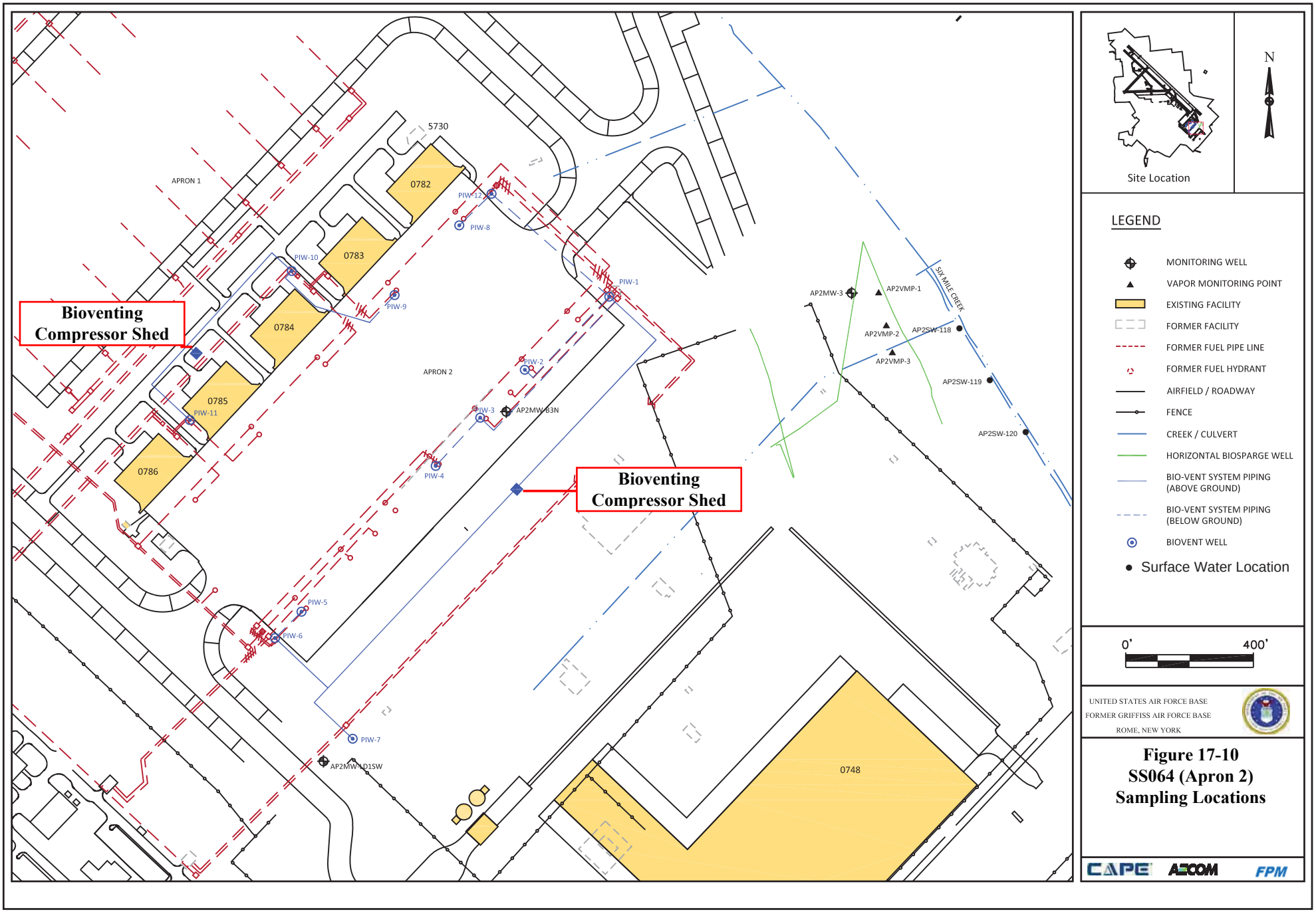

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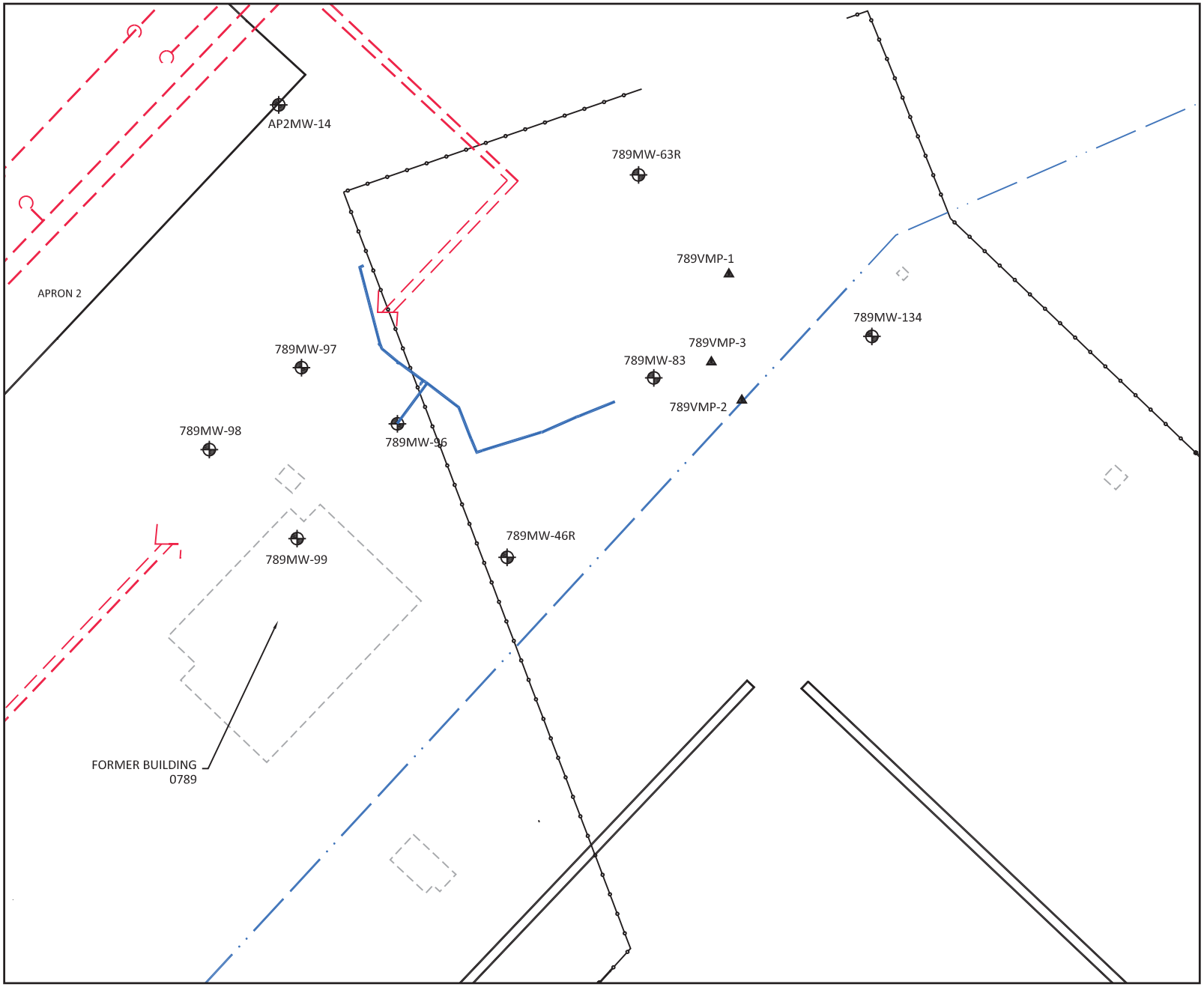

N

Site Location

LEGEND

789MW-63R — MONITORING WELL

789VMP-2 — VAPOR MONITORING POINT

EXISTING FACILITY

FORMER FACILITY

FORMER FUEL PIPE LINE

FORMER FUEL HYDRANT

AIRFIELD / ROADWAY

CREEK / CULVERT

FENCE

VES SYSYTEM

0' 150'

UNITED STATES AIR FORCE BASE
FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-11
SS067 (Building 789)
Sampling Locations

CAPE AECOM FPM

Layout-Sheet Name: 1
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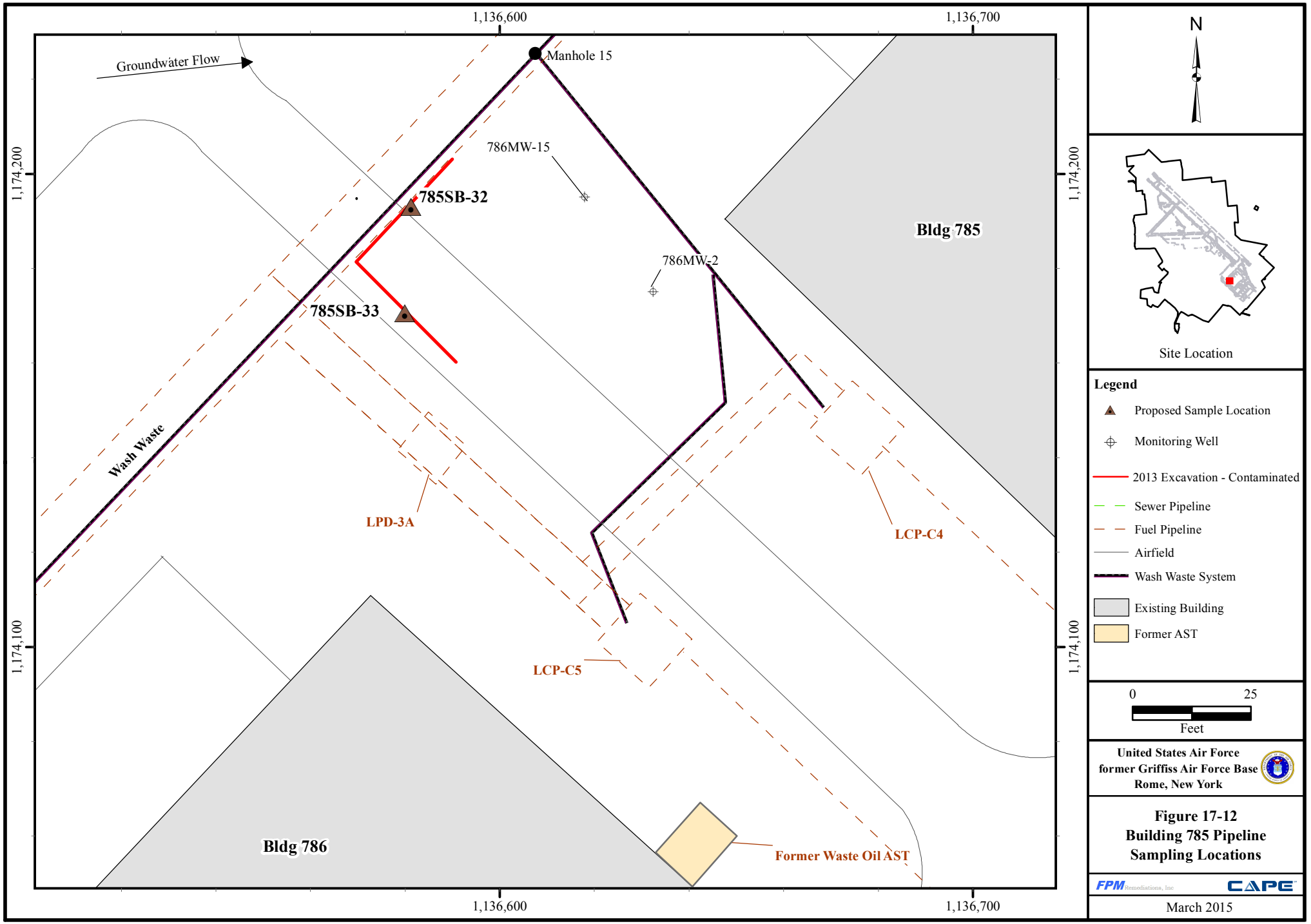

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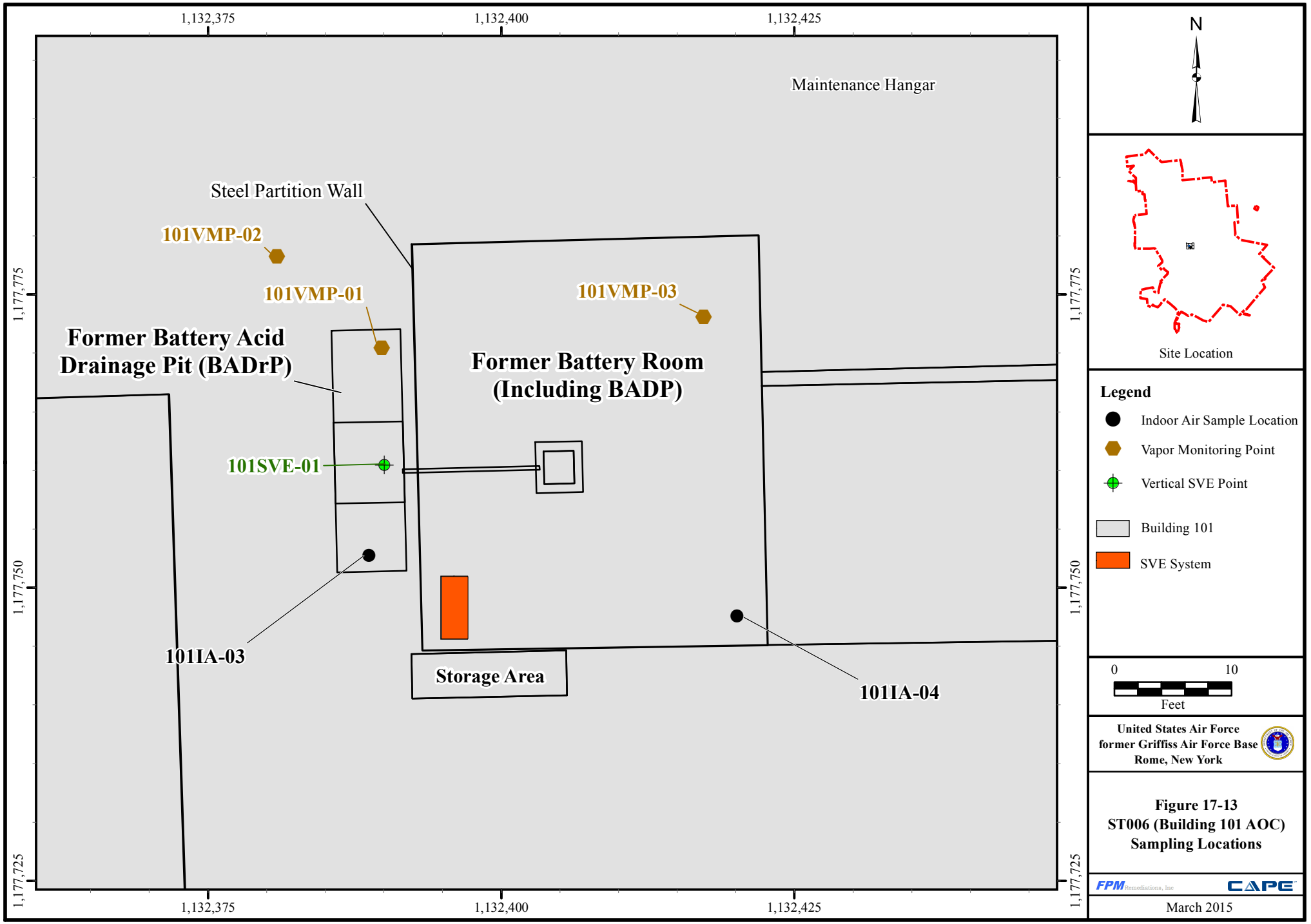

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Appendix A

APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

ATTACHMENTS

1 Sub-Surface Soil Sampling

1.1 Purpose and Scope

This Standard Operating Procedure (SOP) describes the equipment, materials, field procedures, and documentation procedures for collecting sub-surface soil samples using direct push or auger methods for soil characterization and chemical analysis.

Health and safety procedures and equipment to be used during soil sampling are described in a separate site-specific Site Safety and Health Plan (SSHP). These SOPs are intended to be used with the former Griffiss AFB Uniform Federal Policy Quality Assurance Project Plan (UFP QAPP), the existing former Griffiss AFB Field Sampling Plan (FSP) and with other SOPs listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

1.2 Equipment and Materials List

One of the following drilling equipment:

Direct push rig (e.g., Geoprobe® rig or similar) with appropriate drilling and sampling tools (sub-surface soil)

Hollow Stem Auger Kit and electric drill

Hand Auger

The following equipment and materials should be on site for sub-surface soil sampling regardless of the drilling equipment used:

Photoionization Detector (PID) (with 10.2 eV lamp)

Weighted tape measure and ruler with 0.01-foot increments

Surveyor's stakes and flags

Field logbook

Drilling Log form

Sample Collection Field Form

Stainless-steel bowl and spoon

Sample containers

Sample container labels

Label tape (clear)

Ziploc® bags

Paper towels

Digital Camera

Waterproof and permanent marking pens

Plastic sheeting

Trash bags

Ice chest with ice

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

Granular bentonite and potable water

1.3 Locating the Sampling Points

The facilities designated for sampling are shown on figures provided in the UFP-QAPP (Worksheet #17). The approximate soil sampling locations will be identified on site figures before field work commences. The exact soil sampling locations will be determined in the field. Sampling coordinates will be mapped on the front of the Drilling Log in the Location Sketch/Comments Area. The sampling locations will be defined in the investigation specific work plan similar to previous investigation and long term monitoring locations.

When each soil sampling location is identified in the field, the sampling point identification will be entered in the field logbook and on the Drilling Log. Include any information concerning nearby landmarks, or other information that will help to re-locate the point in the future. Mark the sample locations using surveyor's stakes and flags, and label the flag using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid out to identify locations and tie the locations to site landmarks (such as foundations) if available. If the surveyor's stake is offset from the sample location, the offset will be noted on the field map and the field logbook.

1.4 Soil Sampling Procedures

At several sampling sites, the sampling locations may be in concrete or asphalt covered areas. Therefore, at these locations, cores will be drilled through the concrete or asphalt at areas most likely to contain contamination (significant cracks or low points). Direct push technology will be utilized after the concrete has been cored. Direct push samples will be collected using a dual tube sampling system or a discrete interval, piston-type sampler (Geoprobe®, MacroCore®, or equivalent). With a dual tube system, the outer rods remain in the ground while the inner rod and sample liner are extracted to retrieve a soil sample from the desired interval. Soil samples may be collected continuously throughout the depth of the direct push boring or from discrete intervals. The direct push rods will be decontaminated between boring locations, but not between samples at the same boring since a new acetate liner is used for each sample.

With a piston-type sampler, a four-foot or five-foot-long stainless steel sampler with an acetate liner is advanced to the top of the desired sampling interval. The sampler is closed to soil during advancement of the sampler to the desired sampling interval. When the top of the desired sampling interval is reached, a piston rod inside the sampler is unlocked through the drill rods,

and the sampler is advanced to the bottom of the sampling interval. The sampler and all drill rods are then removed from the ground, and the acetate liner is removed from the piston sampler. Aside from the cutting shoe, the soil sampler never comes in contact with the soil sample. The cutting shoe is decontaminated after each sample collected, and a new acetate liner is used for every sample interval. The outer sampling barrel is decontaminated after each boring is completed. The sampling will be documented in the field logbook and drill log.

With a hand auger or hollow stem auger kit, the auger head will be advanced manually to the depth. Auger extensions will be used when sampling at depths exceeding 4 feet. Once the desired depth is achieved, the auger is removed for sample collection as described below. Following collection, the hand auger or hollow stem auger kit will be decontaminated. When using manual samplers, the sampling will be documented in the field logbook and Soil/Sediment sampling form.

At each sampling location, the sampler will be advanced by a combination of hydraulic vertical pressure and percussion hammering. Once the target depth is achieved, the sample will be withdrawn and the liner filled with the soil sample is retrieved.

The following procedures will be followed once the soil sample has been retrieved:

Don a clean pair of nitrile gloves.

Cut acetate sleeve to provide access to the soil sample (direct push sampling only).

Measure the recovery. Record the sampling interval and recovery on the drilling log.

Remove soil smear from the outside of the acetate sleeve and examine the sample, with particular attention for visible evidence of staining, odors, or other evidence of contamination. Record the soil description on the Drilling Log or Soil/Sediment Sampling Form.

Conduct PID screening of the soil. The soil with the highest PID levels will be collected for a sample.

The soil from the sampling interval will be removed from the liner and homogenized in a stainless-steel bowl. Once the soil has been homogenized, fill the appropriate sample containers as specified in the UFP - QAPP (Worksheet #19). Record the sample interval and analysis requested on the Drilling Log or Soil/Sediment Sampling Form and the chain of custody (COC).

Label, store, transport, and document the samples (depending on the use of the sample) according to SOP No. 7. The parameters for analysis and preservation are specified in UFP QAPP Worksheet #19.

If no other samples will be collected from the boring, abandon the boring by backfilling the hole with hydrated granular bentonite. Pour the granular bentonite down the hole in approximate 1-foot to 2-foot lifts, and then pour approximately 0.5 gallon of potable water down the hole to hydrate the bentonite. Continue this from the bottom of the hole to the surface.

1.5 Field Quality Assurance/Quality Control Samples

Field quality assurance/quality control (QA/QC) samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample

collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

1.5.1 Field Blanks

Field blanks are QC samples collected to evaluate potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during volatile organic compounds (VOCs) sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.).

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

1.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then semi-volatile organic compounds (SVOCs), including polynuclear aromatic hydrocarbons (PAHs); metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

1.5.3 Matrix Spikes and Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the

containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample containers will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

1.6 Field Documentation

Field documentation for sub-surface soil sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Two forms are used in the field during sub-surface soil sampling. These forms include the Drill Log and the Soil/Sediment Sampling Form. Each form is described in Section 1.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss.

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 7.

1.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

1.6.2 Field Forms

<u>Drill Log</u>

The Drilling Log contains the following minimum information:

Project name and number

Contractor company, field personnel

Boring Identifier

Drilling subcontractor company and name of drilling personnel

Site Identifier

Brand and model of drill rig

Sizes and types of drilling and sampling equipment

Surface elevation (if available, this may be entered later after the survey)

Date drilling started and finished

Overburden thickness, depth drilled into rock, and total depth of hole

Depth to water during drilling and depth to water after drilling with elapsed time

Number of geotechnical samples, type of samples, and core boxes (if cores are saved)

Number of chemical samples and requested analyses

Signature of field geologist who completed the Drilling Log field form

Field sketch showing the boring location

Sampling interval and measured sample recovery.

A description of the recovered soil sample in accordance with the Unified Soil Classification method for unconsolidated geologic materials. The descriptions should include origin, grain size, sorting, texture, structure, bedding, color, moisture content, and consistency.

Sample Identifier

Sample Collection Time

As applicable, field screening results, geotechnical samples, chemical samples, and blow counts (split-spoon sampling only).

As applicable, record pertinent observations (such as odors, staining, colors, changes in drill rod advancement, chatter, water, etc.) in the "Remarks" column.

If portions of the Drilling Log are not applicable (e.g., if samples are not collected for chemical analysis or if cores are not collected, etc.) record an "NA" in the appropriate location on the form.

Bore hole abandonment (method of abandonment)

Soil/Sediment Sampling Form

The Soil/Sediment Sampling Form contains the following minimum information:

Field personnel

Project name and number

Site Identifier

Sample Location Identifier

Sizes and types of sampling equipment

Date of sample

Sampling depth.

A description of the recovered soil sample. The descriptions should include origin, grain size, texture, structure, color, and odor.

Comments or Observations
Sample Identifier
Sample Collection Time

2 Groundwater Sampling

2.1 Purpose and Scope

This section defines the SOP for the collection of groundwater samples at the former Griffiss AFB. This procedure describes equipment, field procedures, and QA/QC procedures necessary to collect groundwater samples. The sample locations and frequency of collection are specified in the project UFP QAPP.

This SOP is intended to be used together with the UFP QAPP and other appropriate SOPs. Health and safety procedures and equipment that will be required during the investigation are detailed in the SSHP. Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

2.2 Equipment and Materials List

The following equipment will be used during well purging and sampling:

Bailer Sampling:
Well lock keys (if required)

Water level probe with 0.01-foot intervals

Assorted tools (knife, screwdriver, etc.)

Disposable bailers

Nylon rope

Multi-parameter water quality meter (Horiba U-52, YSI 556, or similar)

Calibration fluids

Plastic squeeze or spray bottle filled with de-ionized water

Plastic or glass container (for field parameter measurements)

Paper towels

Calculator

Field logbook

Waterproof and permanent marker

Appropriate containers for holding purged water

Appropriate health and safety equipment, as specified in the SSHP

Well purging and sampling form for bailer sampling

Appropriate decontamination supplies, as specified in SOP No. 8.

Cooler with ice

Garbage bag

Sample labels

Sample bottles with preservatives added will be obtained from the analytical laboratory

Several extra sample bottles in case of breakage or other problems

Low Flow Sampling:

Well lock keys (if required)

Water level probe with 0.01-foot intervals

Assorted tools (knife, screwdriver, etc.)

QED MP10 micropurge digital controller (or similar)/ Well wizard® 3020 oil-less battery powered electric compressor (or similar)

Marine battery

Multi-parameter water quality meter (Horiba U-52, YSI 556, or similar)

Polyethylene tubing (assorted diameters)

Flow-through cell

Plastic, see-through measuring cup (2 cups size)

Calibration fluids

Plastic squeeze or spray bottle filled with de-ionized water

Polyethylene (PE) or glass container (for field parameter measurements)

Paper towels

Garbage bags

Calculator

Field logbook

Waterproof and permanent marker

Appropriate containers for holding purge water

Appropriate health and safety equipment, as specified in the SSHP

Well purging and sampling form for low-flow sampling

Appropriate decontamination equipment, as specified in the SOP No. 8

Cooler with ice

Sample labels

Sample bottles with preservatives added will be obtained from the analytical laboratory

Several extra sample bottles in case of breakage or other problems

2.3 Identifying the Groundwater Sampling Locations

The groundwater sampling locations will be identified in the site-specific work plan (WP). All existing monitoring wells have been surveyed by a certified surveyor and included on maps and figures. All additional monitoring wells will be surveyed after well completion and development.

2.4 Groundwater Sampling Procedures

This section summarizes the step-by-step procedures for collecting groundwater samples in the field. Observations made during sample collection will be recorded in the field notebook and on the well purging and sampling form.

The purpose of well purging is to remove stagnant water from the well and obtain representative water from the geologic formation while minimizing disturbance to the collected samples. Before a sample is collected, the well will be purged until field parameters have stabilized or until the well is pumped or bailed dry. Evacuated groundwater shall be contained for proper disposal if the groundwater is significantly impacted (i.e., heavy sheen or free product), and necessary precautions shall be taken to prevent spilling of water. The following Sections 2.4.1 and 2.4.2 detail sample collection using the bailer collection method and low flow collection methods.

2.4.1 Bailer

Before well purging begins, the following procedures will be performed at each well:

The condition of the outer well casing, concrete well pad, and any unusual conditions of the area around the well will be noted in the field logbook.

The well will be opened.

The condition of the inner well cap and casing will be noted.

The depth of static water level and total well depth will be measured (to nearest 0.01 foot) and recorded from a measuring point on the well casing. The measuring point should be identified, and time indicated in the field logbook.

The volume of water in the well casing will be calculated in gallons based on water column height and casing diameter. Three casing volumes will be calculated.

An initial sample will be obtained for field measurements of temperature, pH, conductivity, turbidity, dissolved oxygen (DO), and oxygen reduction potential (ORP) and for observation of water quality. These measurements will also be used during the evaluation of chemical analytical data.

Three water volumes will be purged. Temperature, pH, conductivity, turbidity, DO, and ORP measurements will be recorded at a minimum of one set of readings per well casing volume purged to determine whether the water chemistry has stabilized. Generally, pH values within $\pm$0.1 pH unit, temperature within $\pm 1^{o}$C, and conductivity within $\pm$ 5% milli-siemens per centimeter (ms/cm) between three consecutive readings indicate adequate stability of the water

chemistry. If the parameters are not stable, purging will continue, measuring pH, temperature, and conductivity after each one-half well volume.

If the well is bailed dry during evacuation, it will be assumed that the purpose of removing 3 well volumes of water has been accomplished, that is, removing all stagnant water which had prolonged contact with the well casing or air. If recovery is very slow, samples may be obtained as soon as sufficient water is available.

The following sampling procedure is followed when using disposable bailers:

Typically, new disposable equipment (PE bailer and nylon rope) are used for each sampling location. Decontaminated sampling equipment will be assembled if necessary.

All sample bottles for all analyses will be gathered and identification labels for each sample bottle will be completed for each sample and affixed to the bottles.

The bailer will be lowered **slowly** and **gently** into contact with the water in the well. The well will be checked for light and dense NAPL. After checking for the presence of NAPL, the bailer will be lowered to the same depth in the well each time.

The bailer will be retrieved **smoothly** and the water will be **slowly** drained into the sample containers through the bailer's bottom discharge control device.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

The bailer and string will be removed from the well and placed in garbage bags for proper disposal as household waste.

The well cap will be replaced and locked.

Field documentation will be completed, including the COC.

2.4.2 Low Flow

Before well purging begins, the following procedures will be performed at each well:

The condition of the outer well casing, concrete well pad, and any unusual conditions of the area around the well will be noted in the field logbook.

The well will be opened.

The condition of the inner well cap, casing and associated tubing will be noted.

The depth of static water level will be measured (to nearest 0.01 foot) and recorded from a measuring point on the well casing, the measuring point should be identified, and time indicated in the field logbook.

The low flow equipment is set-up at the well. The set-up includes:

Connect the Well Wizard® compressor to the marine battery.

Connect air hose from MP10 controller and the Well Wizard® compressor and air hose from the MP10 controller to the air intake of the dedicated bladder pump.

Connect multi-parameter water quality meter with flow-through cell will be connected with new disposable PE tubing to the dedicated bladder pump and associated tubing.

The water purge rate will be set within the range of 100 to 500 ml per minute.

During water purging, water will flow through the multi-parameter water quality equipment flow-through cell. Temperature, pH, conductivity, DO, ORP, and turbidity measurements will be recorded until the parameters have stabilized. Measurements will be collected for each flow-through cell volume. Stabilization parameters are: pH values is $\pm$0.1 pH unit, conductivity values is $\pm$ 3% mS/cm, turbidity is $\pm$ 10% nephelometric turbidity units (NTU) or general turbidity readings below 50 NTU, dissolved oxygen is $\pm$ 10% milligrams per liter (mg/L), and ORP is $\pm$ 10 milliVolts (mV) for three consecutive measurement events. If the parameters are not stable, purging will continue for a maximum of one hour.

The following sampling procedure is to be used when using the low-flow method:

All sample bottles for all analyses for the sampling locations are organized and identification labels for all sample bottles are completed.

The discharge PE tubing will be unhooked from the flow-through cell.

Groundwater samples are collected with water purge rates at or below 250 ml/min.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

The sampling equipment is turned off and disconnected from the well.

The well cap will be replaced and locked.

Field documentation will be completed, including the COC.

2.5 Field Quality Assurance/Quality Control Samples

The well sampling order will be dependent on expected levels of contamination in each well, if known, and will be determined prior to sampling. Typically, the sampling order of the monitoring wells is from the least contaminated well to the most contaminated well. QA/QC samples will be collected during groundwater sampling.

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples are labeled with QA/QC identification numbers and sent to the laboratory in the same batch as the normal samples for analyses.

2.5.1 Field Blanks

Field blanks are QC samples that check for potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during VOCs sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.).

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

2.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then SVOCs, including PAHs; metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

2.5.3 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample container will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

2.6 Field Documentation

Field documentation for groundwater sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Two forms are used in the field during groundwater sampling. These forms include the Bailer Sampling Form and the Low-Flow Sampling Form. Each form is described in Section

2.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss. The field forms will also be provided in the Daily Chemical Quality Control Reports (CQCR).

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, COCs, used during the sampling event are detailed in SOP No. 7.

2.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

2.6.2 Field Forms

<u>Bailer Sampling Form</u>

The Bailer Sampling Form contains the following minimum information:

Project name and number

Sampling personnel

Site Identifier

Date of sample

Well number

Well Diameter

Weather conditions

Depth to water and total depth of well

Purge volume calculations

Purge date

Purge method

Water characteristics and appearance

Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)

Sample ID

Sample time
Any QA/QC Samples

Low-Flow Sampling Form

The Low-Flow Sampling Form contains the following minimum information:

Project name and number
Sampling personnel
Site Identifier
Date of sample
Well number
Well Diameter
Weather conditions
Depth to water and total depth of well
Pump intake depth
Depth of water during purging
Purge date
Purge method
Water characteristics and appearance
Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)
Purge rate
Sample ID
Sample time
Any QA/QC Samples

3 Surface Water Sampling

3.1 Purpose and Scope

The purpose of this section is to define the SOP for collecting surface water samples at the former Griffiss AFB. This SOP describes the equipment, field procedures, and QA/QC procedures implemented for the using the hand tools for collecting grab surface water samples.

This SOP is intended to be used together with the FSP and other appropriate SOPs. Health and safety procedures and equipment for the investigation are detailed in the project SSHP.

Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

3.2 Equipment and Materials List

The following equipment and materials should be on site for surface water sampling:

Long-handled stainless steel sample cup

Multi-parameter water quality meter (Horiba U-52 or YSI 556 or similar)

Surveyor's stakes and flags

Field logbook

Field Sampling Forms

Container to hold water for water chemistry parameter measurements

Stainless steel surface water grab sampler (long handled stainless steel cup)

Sample containers

Sample container labels

Label tape (clear)

Ziploc® bags

Paper towels

Digital camera

Waterproof and permanent marking pens

Trash bags

Ice chest with ice

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

3.3 Locating the Sampling Points

The surface water sampling locations will be identified in the site specific WP and will be identical to current LTM sample locations. These locations have been plotted on sampling location maps for each site.

3.4 Surface water Sampling Procedures

The following procedures will be followed to collect surface water samples:

Decontaminate sampling equipment according to SOP No. 8.

Don a clean pair of nitrile gloves.

Collect surface water using grab sampler and place water into container for water chemistry parameters. The sample for these parameters will be collected while minimizing disturbance to the surface water or sediment in the creek.

Water quality measurements will be collected with a multi-parameter water quality system before sampling and will include pH, temperature, specific conductance, dissolved oxygen, ORP, and turbidity at each surface water sampling location. Measurements will be recorded on the field sampling form immediately.

After water quality parameter collection, the surface water sample will be collected using a grab sampler. Previously preserved sample bottles will be filled by pouring sample water from this cup.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

3.5 Field Quality Assurance/Quality Control Samples

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

3.5.1 Field Blanks

Field blanks are QC samples that check for potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during VOCs sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.)

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

3.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then SVOCs, including PAHs; metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

3.5.3 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample container will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

3.6 Field Documentation

Field documentation for surface water sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Field forms used during the surface water sampling include the Bailer Sampling Form. This form is described in Section 3.6.2 and was used for surface water sampling in previous LTM sampling rounds. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss. The field forms will also be provided in the Daily Chemical Quality Control Reports (CQCR).

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 7.

3.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

3.6.2 Field Forms

<u>Bailer Sampling Form</u>

The Bailer Sampling Form used during the surface water sampling contains the following minimum information:

Project name and number

Sampling personnel
Site Identifier
Date of sample
Sample location number
Weather conditions
Collection method
Water characteristics and appearance
Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)
Sample ID
Sample time
Any QA/QC Samples

4 Soil Vapor Sampling (indoor, outdoor, and sub-slab vapor)

The purpose of this section is to define the SOP for collecting soil vapor samples at the former Griffiss AFB using electrical drills and soil vapor probes. This SOP describes the equipment, field procedures, and QA/QC procedures implemented for soil vapor sampling.

The sampling methodologies provided below were adapted from the NYSDOH SVI guidance document (NYSDOH, October 2006). Site-specific details and modifications have been implemented through the Sub-Slab Vapor Mitigation Design Work Plan.

Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

4.1 Equipment and Materials List

The following equipment and materials should be on site for soil sampling:

Summa® canisters, minicans, or similar

PID (ppbRAE or similar)

Regulator for vapor sample canister preset to the appropriate sample duration

Vacuum pump (manual or electric)

Stainless steel or PE vapor implants with 'speedfit' push fitting

PE tubing

Box cutter

Tee's for duplicate sample collection

Field logbook

Field Sampling Forms

Digital camera

Waterproof and permanent marking pens

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

4.2 Locating the Sampling Points

The indoor, outdoor, and sub-slab vapor sample locations will be predetermined in accordance with the site-specific sampling WP.

4.3 Soil Vapor Sampling Procedures

4.3.1 Soil Vapor Sampling

4.3.1.1 Temporary Soil Vapor Probe Installation and Abandonment

The installation and abandonment procedure is as follows:

- A Geoprobe® shall be employed to attain a depth of at least 5 ft below ground surface (bgs) for each soil vapor probe. A 2.5-inch coring machine shall be used to core through the concrete prior to engaging the Geoprobe. If necessary; a hollow-stem auger can be used to attain the desired depth;
- Once the target depth is reached, the rods will be pulled up one foot, exposing the void space, and the sampling apparatus will be set up in the borehole;
- New ¼-inch laboratory grade polyethylene tubing equipped with a threaded stainless steel fitting will be attached to a disposable soil vapor drive point to prevent infiltration of the atmospheric air present at land surface directly above the soil boring (ambient air);
- A clay seal will then be placed at land surface in the annular space between the Geoprobe® rods and the concrete surface, as well as between the tip of the rods and the sample tubing;
- The sampling tubing will be connected to a 'T' connector three-way valve assembly, with one end of the 'T' connector leading to a vacuum pump and the other end leading to a pre-evacuated summa canister with a calibrated regulator;
- The soil vapor sample tubing will then be purged of approximately two volumes of the sample tubing using a vacuum pump set at a rate of approximately 0.2 liters per minute;
- After sampling is completed, the borehole shall be abandoned by being tremie grouted to land surface using a bentonite grout.

4.3.1.2 Soil Vapor Sample Collection

The sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- Prior to formal sample collection, a tracer gas (i.e., helium) shall be used to verify the integrity of the soil vapor probe seal. To do so:
  - ✓ The immediate vicinity of the area where the probe intersects the ground surface shall be exposed to tracer gas using a garbage bag, cardboard box, or plastic pail;
  - ✓ At least one implant volume (i.e., the volume of the sample probe and tube) shall be purged using a flow rate of not more than 0.2 L/min;
  - ✓ Using the same flow rate as the purge (i.e., less than 0.2 L/min), a vapor sample shall be collected from the probe using a Tedlar bag;
  - ✓ The Tedlar bag shall be fitted with a portable monitoring device (i.e., a Gas Check 3000 meter, which measures the rate of the helium leakage at the land surface) and screened for helium. The enriched area (i.e., within the garbage bag/cardboard box/plastic pail) will also be screened for helium.

- ✓ If the concentration of helium is greater than 20% of the helium detected in the enriched area, the seal is not adequate and should be reset. The sample rods will be purged again until the helium is no longer detected at levels greater than 20% of the enriched area located directly above the borehole.

- Once the integrity of the seal has been verified, to ensure samples collected are representative, three implant volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the sample;
- Flow rates for both purging and collecting shall not exceed 0.2 L/min to minimize outdoor air filtration during sampling;
- Following the purging, the valve leading to the pump will be closed, the pump will be turned off, and the soil vapor will be directed to a 100% certified 1-L Summa® canister provided by the laboratory. The sample shall be collected using the canister's regulator to restrict the sample collection rate.
- After sample collection, the soil vapor will be screened using a photoionization detector (PID), calibrated daily with a 100 parts per million (ppm) isobutylene standard.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, and any relevant observations such as odors and readings from field instrumentation.

4.3.2 Sub-slab Vapor Sampling

4.3.2.1 Temporary Sub-slab Vapor Probe Installation and Construction

As noted in the NYSDOH guidance document, during colder months, heating systems should be operating at least 24 hours prior to and during the scheduled sampling time to maintain normal indoor air temperatures. Prior to installation of the sub-slab vapor probes, the building floor should be inspected and any penetrations (i.e., cracks, floor drains, utility perforations, sumps, etc.) should be noted and recorded. Probes should be installed at locations where the potential for ambient air infiltration via floor penetrations is minimal.

The installation procedure is as follows:

- A rotary hammer drill will be used to create 1-inch diameter holes through concrete and into sub-slab material (e.g., sand or sand and gravel). Drilling into sub-slab material will create an open cavity to prevent obstruction of probes by small pieces of gravel;
- Probes will be constructed from dedicated ¼ inch-diameter laboratory grade polyethylene tubing;
- Tubing shall not extend further than 2 inches into the sub-slab material;
- The implant shall be sealed to the surface with permagum grout, melted beeswax, putty, or other non-VOC-containing and non-shrinking product;
- After sampling is completed, the borehole shall be abandoned in accordance with the procedures described in Section 5.5.3, in the UFP QAPP for Performance Based-Remediation at the Former Griffiss AFB (CAPE/FPM, November 2011).

4.3.2.2 Sub-slab Vapor Sample Collection

The sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- To ensure samples collected are representative, three implant volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the sample;
- Flow rates for purging shall not exceed 0.2 L/min to minimize outdoor air filtration during sampling. Purge air shall be collected in a Tedlar bag so it is not released into the building;
- Samples shall be collected over an 24-hour time period, consistent with concurrent indoor and outdoor air samples, if possible;
- Samples shall be collected in 100% certified 6-L Summa® canisters provided by the laboratory.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation.

In addition, floor plan sketches should be drawn that include the floor layout with sample locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north) and any other pertinent information. If possible, photographs should accompany floor plan sketches.

4.3.3 Indoor/Outdoor Air Sampling

4.3.3.1 Pre-sampling Inspection and Documentation

As noted in the NYSDOH guidance document, during colder months, heating systems should be operating at least 24 hours prior to and during the scheduled sampling time to maintain normal indoor air temperatures. Prior to collecting indoor air samples, a pre-sampling inspection should be performed prior to each sampling event to identify conditions that may affect or interfere with the proposed testing. The inspection should evaluate the type of structure, floor layout, physical conditions, and airflows of the building(s) being studied. The inspection information should be identified on the attached Indoor Air Quality Questionnaire and Building Inventory form. In addition, potential sources of chemicals of concern should be evaluated within the building by conducting a product inventory.

In addition, floor plan sketches should be drawn that include the floor layout with sample locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north) and any other pertinent information should be documented. If possible, photographs should accompany floor plan sketches.

Finally, outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sample locations, the location of potential interferences (e.g., gasoline stations, factories, other facilities, lawn mowers, etc.), compass orientation (north), footings that create separate foundation sections, and paved areas. Significant activities in the vicinity of the sample locations (e.g., operation of heavy equipment) should be recorded.

4.3.3.2 Indoor/Outdoor Air Sample Collection

Indoor air samples shall be collected in the vicinity of the sub-slab samples from a height above the ground to represent the breathing zone when occupants normally are seated (i.e., 5 ft.). The locations of the outdoor samples shall be chosen from areas away from wind obstructions, and at a height above the ground to represent the breathing zone (i.e., 3 to 5 ft.).

For either indoor or outdoor air samples, the sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- Samples should be collected during normally occupied periods to be representative of typical exposure;
- Sample collection intakes should be located to approximate the breathing zone for building occupants (i.e., 5 feet above the floor level where occupants are normally seated);
- To ensure that an air sample is representative of the conditions being tested and to avoid undue influence from sampling personnel, samples should be collected for a period of twenty-four (24) hours, and personnel should avoid lingering in the immediate area of the sampling device while samples are being collected;
- The sampling team members should avoid actions (e.g., fueling vehicles, using permanent marking pens) that can cause sample interference in the field;
- Flow rates for collecting samples shall not exceed 0.2 L/min to be consistent with concurrent sub-slab sampling;
- Samples shall be collected in 100% certified 6-L Summa® canisters provided by the laboratory; and
- Indoor and outdoor samples should be collected simultaneously;
- Ideally, samples shall be collected over the same period of time as concurrent sub-slab samples.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation.

4.4 Field Quality Assurance/Quality Control Samples

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

4.4.1 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. A nylon T-barb will be installed in the PE tubing to allow for sampling of one airstream from one sampling point with two vapor sample canisters simultaneously. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

4.4.2 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analysis are used to assess the potential for matrix effects. The MS/MSD sample will be collected from a randomly selected normal sample by the lab. Following the normal analysis, the lab spikes the normal sample canister with the matrix spike and analyses the air in the canister. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

4.5 Field Documentation

The most important aspect of field documentation is thorough, organized, and accurate record keeping. This includes proper preservation and storage of all field documentation. Field documentation for sub-slab vapor sampling includes field logbooks and field forms. The field forms, described in section 6.5.2, include the sub-slab vapor probe monitoring form, indoor/outdoor air monitoring form, weather observation form, and the NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health form.

4.5.1 Field Logbook

All information pertinent to sub-slab sampling will be recorded in a bound field logbook with consecutively numbered pages. The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

4.5.2 Field Forms

<u>Sub-slab Probe Monitoring Form</u>

The Sub-slab Probe Monitoring Form contains the following minimum information:

Date

Time

Sample identification

Sample depth

Field personnel

Instruments

Tracer gas identified and concentration

Sample purge volume

Volume of soil vapor extracted

Summa canister: vacuum before sampling and vacuum after sampling

Apparent moisture content

Comments and observations during sampling

Weather conditions, including the outdoor temperature, barometric pressure, precipitation, ventilation conditions, heating system active?, and windows closed

Indoor/Outdoor Air Monitoring Form

The Indoor/Outdoor Air Monitoring Form contains the following minimum information:

Date

Time

Sample identification

Sample height

Field personnel

Instruments

Type of sample

Duration of air sampled

Volume of sample

Summa canister: vacuum before sampling and vacuum after sampling

Comments and observations during sampling

VOCs used during normal operations of facility

Weather conditions, including the outdoor temperature, barometric pressure, precipitation, ventilation conditions, heating system active?, and windows closed

Weather Observation Form

The Weather Observation Form contains the following minimum information:

Location

Date

Field Personnel

Instruments

Time

Conditions collected prior to sampling, mid day, and end of sampling include:

Precipitation

Atmospheric pressure

Temperature

Wind speed

NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health Form

The NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health Form contains the following minimum information:

Preparer's name

Date/Time

Preparer's affiliation

Phone number

Field Personnel

Occupant

Name

Address

Phone Number

Number of occupants in building and age

Owner or landlord

Name

Address

Phone Number

Building Characteristics

Type of Building

Property type

Multiple units

Air flow

Basement and Construction Characteristics

Heating, Venting, and Air Conditioning information

Occupancy

Factors that may influence indoor air quality
Water and sewer information
Relocation information
Floor Plans
Outdoor plot
Product inventory form

5 Sample Handling, Documentation, and Tracking

5.1 Purpose and Scope

This SOP describes the procedures for sample handling, documentation, and tracking. This SOP is intended to be used with the UFP-QAPP, FSP and with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 4, Soil Vapor Sampling

SOP No. 7, Monitoring Well Installation and Development

5.2 Sample Identification

The sampling locations, sample types, and naming conventions will be established prior to field activities. On-site personnel will obtain assistance in defining any special sampling requirements from the FPM Project Manager or designated Task Manager. Each sample will have a discrete, alpha-numeric sample identification (ID). A unique sample ID is needed to track each sample during the life of this project. In addition, the sample IDs will be used in the database to identify and retrieve the analytical results received from the laboratory. Each sample ID will be assigned at the time of sampling.

Sample ID

The sample ID will be designated as follows: Site Code, Sample Type and Sampling Location Indicator, Sample Depth Identifier, and Sample Type Qualifier.

Site Code

The first segment consists of two to five alphanumeric characters that designate the site code. Site codes for monitoring wells named in previous Griffiss AFB sampling efforts (Law, 1996; FPM, 2001) are listed below:

- LF1 Landfill 1

For the sample designated “LF1M0213AA”, the “LF1” indicates that the site from which the sample was obtained, is the Landfill 1 AOC Site.

Sample Type and Sampling Location Indicator

The second segment consists of one or two alphanumeric characters that indicate the sample type and sampling location indicator. Sample types are as shown below:

- M Groundwater from monitoring well sampling locations

- T Groundwater from direct-push groundwater samples that were not completed as permanent monitoring wells (i.e., temporary well point)
- SW Surface water sample
- SD Sediment sample
- SS Soil Sample
- FS Fish Tissue Sample
- IA Indoor Air
- OA Outdoor Air
- SSV Sub-slab Vapor

The two-digit number following the sample indicator completes the identification of the sampling location at a specific site. For example, for the sample "LF1M0213AA", the "M" indicates that the sample was groundwater taken from a monitoring well, and the "02" indicates that this sample was taken from monitoring well LF1MW-02.

Sample Depth Identifier

The third segment consists of two numerical characters that will be used to identify the depth in feet below TOIC the sample was taken. For the sample designated "LF1M0213AA", the "13" indicates that the sample was obtained at a depth of 13 feet below TOIC.

Sample Type Qualifier

The fourth segment is two alphabetic characters used to designate the type of sample. The first letter denotes the round of sampling completed (e.g., "A" for first quarterly sampling round, "B" for second quarterly sampling round, etc.). The sample types will be identified by the second character as listed below:

- A = Primary sample
- B = Primary sample
- C = Field duplicate groundwater sample
- D = Matrix Spike Duplicate (MSD)
- E = Equipment blank
- F = Ambient blank
- R = Trip blank
- S = Matrix Spike (MS)

The letter A or B appearing at the end of a sample number indicates that the sample is a primary sample. These letters will be selected randomly to mask the predominance of primary samples over QA/QC samples. This system was devised to minimize the likelihood that the laboratory personnel can distinguish the primary samples from the QA/QC samples using the sample identification.

To complete the example, the sample number “LF1M0213AA”, would therefore indicate a primary first-round groundwater sample taken from monitoring well LF1MW-02 at 13 feet below TOIC at the Landfill 1 AOC Site.

5.3 Sample Labels

Sample labels will be completed as much as possible by a designated member of the sampling team prior to beginning field-sampling activities each day. All sample labels will be filled out using waterproof ink. For the pre-designated sampling events (LTM), labels are preprinted by the lab using the COCs developed during sample planning. At a minimum, each label will contain the following information:

Sampler's company affiliation

Site location

Sample ID

Date and time of sample collection

Analyses required

Method of preservation (if any) used

Sample matrix (i.e., soil, groundwater, surface water)

Sampler's signature or initials

5.4 Sample Handling Procedures

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The UFP-QAPP summarizes the information contained in this section and also includes the sample holding times for each analyte.

5.4.1 Sample Containers

Certified, commercially clean sample containers will be obtained from the contract analytical lab. Required preservatives will be prepared and placed in the containers at the laboratory prior to shipment to the site. Appropriate sample containers for the specific analyses required will be listed in the UFP-QAPP (Worksheet #19).

5.4.2 Sample Preservation

Sample preservation efforts will commence at the time of sample collection and will continue until analyses are performed. Samples will be stored on ice at 4°C in coolers immediately following collection. Chemical preservatives, if necessary, will be added to the sample containers by the laboratory prior to shipment to the field, unless otherwise specified in the UFP-QAPP.

5.4.3 Sample Handling and Shipping

The sample containers will be wiped clean of all sample residue and then wrapped in protective packing material (bubble wrap) and taped. Samples will be single-bagged with plastic bags and then placed upright in an iced cooler. A COC form will accompany each cooler.

Coolers will be picked up at the FPM Rome office by the lab courier or shipped by overnight express carrier to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times (UFP QAPP, Worksheet #19). This may require daily shipment of samples with short holding times. The condition of all samples as received and temperature of all coolers will be reported by the laboratory.

5.4.4 Holding Times and Analyses

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest and stability factors, and preservative (if any) used. Allowable holding times are listed in the UFP-QAPP (Worksheet #19).

5.5 Sample Documentation and Tracking

This section describes documentation required in the field notes, on the field sampling forms, on the Daily CQCRs, and on the COCs.

5.5.1 Field Logbook

The purpose of the field log book is to provide a chronological account of all field activities for future reference. Activities logging will be performed to include sufficient information so that the sampling activity can be reconstructed without relying on the memory of field personnel. The logbooks will be kept in the field team member's possession or in a secure place during the investigation. Following the investigation, the logbooks will become a part of the final project file.

All entries in logbooks will be made in waterproof ink and corrections will consist of line-out deletions that are initialed and dated. The following information (as applicable) shall be recorded in the header of the field log book:

Sampler’s printed name and signature

Names of other field personnel (CAPE Team and any CAPE Team subcontractors) and site visitors

Date (month, day, year)

General weather conditions

The following information (as applicable) shall be recorded in the field log book:

Results of equipment calibration

Time and location of sampling (including approximate distance to adjacent landmarks if possible)

Documentation of field measurement results such as total depths and depth to groundwater in monitoring wells.

Sample Identification and time of collection

Any QA/QC sample collected

Decontamination information

Brief discussion of any field decisions, unusual conditions, problems encountered and corrective action taken, and/or changes required by field conditions

Signature and date by person responsible for writing the field notes

In addition to field books, sample forms will also be prepared in the field. The sampling forms will contain the results of any field measurements, sample identification and sampling time. The field measurements included in the sampling form include water chemistry readings. A description of the sampling field forms are included in the sampling matrix specific sections.

5.5.2 Daily Chemical Quality Control Report

Daily CQCRs will be prepared to supplement the information recorded in the field logbook. Daily CQCRs will be prepared by members of the field sampling team and cross-checked for completeness at the end of each day by the sampling team leader and/or Field Manager. They will be signed and dated by individuals making entries. Daily CQCRs will be forwarded to the Quality Assurance Officer for review and approval. The Daily CQCRs will include the following information:

Project name

Project number

Personnel on site

Visitor on site

Subcontractors on site

Weather conditions

Field work performed

Quality control and health and safety activities

Name and title of person completing the Daily CQCR

5.5.3 Chain of Custody

During field sampling activities, traceability of the sample must be maintained from the time that the samples are collected until laboratory data are issued. Information concerning samples collection will be recorded in the field logbook as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form.

The sampler will be responsible for initialing and completing the COC. The sampler will sign the COC when the sampler relinquishes the samples to the lab courier. One COC will be completed daily for the site's samples. The COC will contain the following information:

Sampler's signature and affiliation

Project name

Date and time of collection

Sample ID

Sample type

Analyses requested

Number of containers per sample per analysis

Signature of persons relinquishing custody, dates, and times

Signature of persons accepting custody, dates, and times

Method of shipment

Shipping air bill number (if applicable)

The person responsible for sample shipment to the laboratory will sign the COC form, and retain a copy of the form, document the method of shipment, and send the original copy of the COC form with the samples. Copies of the COC forms documenting custody changes and all custody documentation will be received in the lab packages and kept in the central files. The original COCs will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting.

6 Decontamination

6.1 Purpose and Scope

This SOP describes the equipment, materials, field procedures, and documentation procedures for decontaminating sampling equipment and personnel. The procedures presented below are intended to be used with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 4, Surface Soil and Sediment Sampling

SOP No. 5, Soil Vapor Sampling

SOP No. 8, Monitoring Well Installation and Development

The overall objective of an environmental sampling program is to obtain samples that accurately represent the chemical, physical, and/or biological conditions at the sampling site. Extraneous contaminants can be brought onto the sampling location and/or introduced into the medium of interest during the sampling program (e.g. using sampling equipment that is not properly or fully decontaminated). Trace quantities of contaminants can consequently be captured in a sample and lead to false positive analytical results and, ultimately, to an incorrect assessment of the contaminant conditions associated with the site. Decontamination of sampling equipment (e.g., all non-disposable equipment that will come in direct contact with samples) and field support equipment (e.g., drill rigs, vehicles) is, therefore, required prior to, between, and after uses to ensure that sampling cross-contamination is prevented, and that on-site contaminants are not carried off-site.

6.2 Equipment and Materials List

The following is a list of equipment that may be needed to perform decontamination:

Brushes

Wash tubs

Buckets

Scrapers, flat bladed

Hot water – high-pressure sprayer

Sponges or paper towels

Liquinox® detergent (or equivalent)

Potable tap water

Laboratory-grade de-ionized water

Garden-type water sprayers

Appropriate Health and Safety equipment (i.e., nitrile gloves, safety glasses, etc.)

Appropriate containers for Investigation Derived Waste (IDW).

6.3 Decontamination Procedures

Site activities should be conducted with the general goal of preventing the contamination of personnel and equipment. CAPE Team sampling personnel will bag monitoring instruments, avoid contact with obvious contamination, and employ dust suppression methods as necessary to reduce the probability of becoming contaminated and, therefore, reduce the need and extent of decontamination. However, some type of decontamination will always be required on site.

6.3.1 Decontamination Solutions

A decontamination solution should be capable of removing, or converting to a harmless substance, the chemical of concern without harming the object being decontaminated. The preferred solution is a mixture of detergent and water, which is a relatively safe option compared to chemical decontaminants. A solution recommended for decontaminating consists of 1 to 1.5 tablespoons of Liquinox® per gallon of warm water. Skin should be decontaminated by washing with hand soap and water. The decontamination solution must be changed when it no longer foams or when it becomes dirty. Rinse water must be changed when it becomes discolored, begins to foam, or when the decontamination solution cannot be removed.

6.3.2 Personnel Decontamination

A sample personnel decontamination set-up guideline and equipment and supplies list are included in the SSHP.

6.3.3 Sampling Equipment Decontamination

The following steps will be used to decontaminate sampling equipment:

Personnel will dress in suitable safety equipment to reduce personal exposure as required by the SSHP. Typically for LTM programs, this includes personnel in level D PPE (long pants, long sleeve shirts, steel toe boots, and nitrile gloves).

Gross contamination on equipment will be scraped off at the sampling or construction site with a flat bladed scrape.

Equipment that cannot be damaged by water will be placed in a 5-gallon bucket containing a Liquinox® solution or low-sudsing non-phosphate detergent along with potable water and scrubbed with a bristle brush or similar utensil. Equipment will be rinsed with tap water in a second wash tub followed by a de-ionized water rinse.

Equipment that may be damaged by immersion in water will be carefully wiped clean using a sponge and detergent water and rinsed with de-ionized water. Care will be taken to prevent equipment damage.

Following decontamination, equipment will be placed in a clean area or on clean plastic sheeting to prevent contact with contaminated soil. If the equipment is not used immediately after decontamination, the equipment will be covered or wrapped in plastic sheeting, foil, or heavy-duty trash bags to minimize potential contact with contaminants.

6.3.4 Direct Push Equipment Decontamination

Direct push rigs will be decontaminated at a decontamination station located near the staging area. Direct push rods will be decontaminated at the various drilling locations. The following steps will be used to decontaminate direct push equipment:

The direct push rig will be decontaminated upon mobilization to the site and demobilization from the site. The direct push rods will be decontaminated between each boring location.

Personnel will dress in suitable PPE to reduce personal exposure as required by the SSHP.

Equipment showing gross contamination or having caked-on soil cuttings will be scraped with a flat-bladed scraper at the sampling or construction site.

The direct push rods will be washed with a hot water, high-pressure sprayer then rinsed with potable water. OSHA requires that proper PPE must be worn when operating pressure-washing equipment. A rain suit, boots, hard hat, and a face shield are recommended to be worn. All personnel must be kept out of the path of steam or water spray.

Following decontamination, direct push rods will be placed on a clean area. If the direct push rods are not used immediately, they must be stored in a designated clean area.

6.3.5 Equipment Leaving the Site

Vehicles used for activities in non-contaminated areas shall be cleaned on an as-needed basis, as determined by the Site Safety and Health Officer (SSHO), using soap and water on the outside and vacuuming the inside. On-site cleaning will be required for very dirty vehicles leaving the area or equipment that has been operated in contaminated areas. Drilling and trailers used in contaminated areas will be pressure washed before the equipment is removed from the site to limit exposure of off-site personnel to potential contaminants.

6.3.6 Responsible Authority

Decontamination operations at each hazardous waste site shall be supervised by the SSHO. The SSHO is responsible for ensuring that all personnel follow decontamination procedures and that all contaminated equipment is adequately decontaminated. The SSHO is also responsible for maintaining the decontamination zone and managing the wastes generated from the decontamination process.

6.3.7 Investigation Derived Waste

Liquid wastewater from decontamination will be drummed and properly disposed of. Solid waste, including sample liners and PPE, will be bagged and removed from the site as household waste.

6.4 Emergency Decontamination

Emergency decontamination procedures should be followed if necessary to prevent the loss of life or severe injury. In the case of threat to life, decontamination should be delayed until the victim is stabilized; however, decontamination should always be performed first, when practical, if it can be done without interfering with essential lifesaving techniques or first aid, or if a worker has been contaminated with an extremely toxic or corrosive material that could cause severe injury or loss of life. During an emergency, provisions must also be made for protecting medical personnel and disposing of contaminated clothing or equipment.

6.5 Documentation

Sampling personnel will be responsible for documenting the decontamination of sampling and drilling equipment. The documentation will be recorded with waterproof ink in the sampler's field notebook with consecutively numbered pages. The information entered in the field book concerning decontamination will include the following:

Decontamination personnel

Date and start and end times

Decontamination observations

Weather conditions

IDW handling

7 Monitoring Well Installation and Development

7.1 Purpose and Scope

This section described the SOP for drilling, installation, and development of monitoring wells at the former Griffiss AFB. The step-by-step procedures described herein are sufficiently detailed to allow field personnel to properly install and develop wells. All construction materials methods and details will be consistent with the requirements of the New York State Department of Environmental Conservation (NYSDEC) for well installation.

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This SOP is intended to be used with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 5, Soil Vapor Sampling

SOP No. 6, Sample Handling, Documentation, and Tracking

Health and safety procedures and equipment for the investigation are detailed in the SSHP.

7.2 Drilling and Well Installation Procedures

7.2.1 Equipment and Materials List

This section details the required equipment, drilling and installation procedures, and documentation procedures for installation of groundwater monitoring wells and vertical biosparging points at the former Griffiss AFB.

The following is an equipment list for monitoring well installation:

Hollow-stem auger rig capable of installing wells to the desired depth in the expected formation materials and conditions

Weighted tape measure

Water level probe

PID (with 10.2 eV lamp)

Well casing and well screen

Bentonite pellets

Filter pack sand (16-40 silica sand)

Portland Type I or II Cement and powdered bentonite or high solids bentonite for grouting

High pressure grout pump

Protective well casing with locking cap or flush mount manhole assembly with padlock

Steel guard posts (bollards) for stick-up wells

Inertial Pump (Waterra® pump or similar)

High-pressure steamer/cleanser

Long-handled bristle brushes

Wash/rinse tubs or pails

Liquinox® detergent

Plastic bags (Ziploc®)

Self-adhesive labels

Deionized water

Appropriate health and safety equipment

Log book

Boring log sheets

Well construction logs

Appropriate sample containers

Sample cooler and ice

7.2.2 Drilling Method

An auger section is a section of pipe with flanges welded onto it. Each auger section is referred to as a flight. Flights are typically five feet in length. The flights are linked together as each flight is advanced to the ground surface. Sampling tools and the center bit are advanced through the open pipe. The cutting bit has a finger plug which prevents loose soil from entering the open pipe. A split-spoon sampling device may be lowered inside the drill string and driven through the finger plug and ahead of the cutting bit for an in-situ sample as required. The drill string, therefore, serves as a form of casing because it does not have to be withdrawn each time a sample is collected. For the 2-inch-diameter wells that are planned, a 4-1/4 inch inside diameter HSA will be used.

There are several advantages of HSA boreholes. First, the method is rapid in most unconsolidated, fine- to medium-grained geologic materials. Second, drilling fluids are not used to remove cuttings and, therefore, the in-situ chemical conditions of the borehole are not further degraded by either diluting contaminants with added water or contributing suspended solids from drilling mud used to stabilize the borehole walls in soft materials. Third, HSA flights are easily cleaned and decontaminated. Fourth, the auger flights serve as a form of casing, which allows monitoring wells to be constructed by raising the flights as needed.

If flowing sands are encountered, potable water may be added to the augers to equalize the hydrostatic pressure in the boring. If water is added to the augers or borehole, it must be potable and the quantity used recorded in the field logbook.

7.2.3 Stratigraphic Logging

Borehole stratigraphy will be logged by examining continuous core soil samples or soil cuttings. The data will be recorded on the boring log and will include the following information:

Project name and number

Drilling company name

Date drilling started and finished

Type of bit and size

Casing sizes and depths

Well completion details

Driller's name

Geologist's name

Type of drill rig

Boring number

Surface elevation (if available)

Sample depths and times

Sample characteristics with depth, such as lithology, grain size, sorting, texture, structure, bedding, color, moisture content, and the Unified Soil Classification (if in unconsolidated geologic materials)

Water levels

Geophysical or video log run (if any)

Drilling observations

Other pertinent information

7.2.4 Well Material Specifications

This section describes the well materials to be used for groundwater monitoring well installations.

7.2.4.1 Well Casings

Well casing will consist of new, threaded, flush-joint, 2-inch ID, schedule 40 PVC. O-rings will be used at all joints. Heat-welded joints and/or gaskets will not be used. The tops of all well casings will be fitted with caps (J-plug) which can be easily removed by hand. The well casing will be brought to the site in its factory post-cleaning plastic wrapping and steam cleaned before installation will not be required.

7.2.4.2 Well Screens

Well screens will consist of new, 2-inch ID threaded PVC with factory machined slots. The screen slot sizes are 0.010-inches and 0.020-inches. An end-plug will be placed at the bottom of the screen. The screen depth will intersect the uppermost portion or the saturated zone. All well screens will have an inside diameter equal to or greater than that of the well casing. Well screen length will be 10 feet.

7.2.4.3 Filter Pack

The filter pack material for the monitoring wells will consist of a #16-40 pre-washed environmental grade silica sand or equivalent. For shallow wells, less than 30 ft bgs, the filter pack will be poured into the open boring. For deeper wells, the filter pack will be placed by tremie pipe from the bottom of the borehole to two feet above the top of the screen interval. Surging of the well may be necessary during filter pack placement to obtain an adequate pack placement around the well screen. The depth of the filter pack will be continuously probed with a weighted tape during placement to monitor pack placement and avoid bridging.

7.2.4.4 Bentonite Seal and Annular Seal

A bentonite seal will be installed above the filter pack in the monitoring wells. The seal will consist of a two-foot interval of bentonite chips or pellets placed by gravity feed from the ground surface and will be hydrated prior to placement of the annular seal. The annular seal will be placed by gravity feed from just above the bentonite seal to within three feet of the ground surface and shall consist of cement grout, neat cement, concrete, or bentonite grout.

7.2.4.5 Well Completion

Two well completion types will be used. These include the flush mount and stick-up well completions.

For high traffic areas, flush mount completions will be installed. The flush mount includes a 8-inch OD traffic rated manhole and concrete pad. Following manhole installation, a locking water-tight security plug will be installed on top of the PVC riser. At a minimum the monitoring well identification number and installation date will be stamped or engraved on to the tag.

For areas with no traffic, stick-up completions will be installed. The stick-up completions include a steel 8-inch OD stick-up pipe, traffic bollards, and concrete pad. Following stick-up completion installation, a locking water-tight security plug will be installed on top of the PVC riser. At a minimum the monitoring well identification number and installation date will be stamped or engraved on to the tag.

7.2.5 Well Installation Procedure

The procedure for monitoring well installation using HSA methods is as follows:

1. Decontaminate all well materials (if necessary) according to SOP No. 8. Following decontamination, all personnel that handle the casing will don a clean pair of rubber or nitrile gloves.
2. Measure each joint of casing and screen to nearest 0.10 foot.
3. Assemble screen and casing as it is lowered into the boring or inside the HAS pipe. Attach stainless steel centralizers if required.
4. Lower screen and casing to the bottom of the boring.
5. Record level of top of casing and calculate screened interval. Adjust screen interval by raising or lowering assembly to desired interval if necessary and add sand to raise the bottom of the boring to the base of the screen. A 1.5-inch diameter, 10-foot long pipe may be lowered into the well to check for straightness. If the pipe will not pass the entire length of the well casing, the well will have to be removed and reset or, if this is not possible, a new well will be installed.
6. Begin adding filter pack sand around the annulus of the casing by slowly gravity feeding the sand (through the tremie pipe if required). Repeated depth soundings should be taken to monitor the level of the sand.
7. Allow sufficient time for the filter sand to settle through the water column outside the screen and casing before measuring the sand level.
8. Extend the filter pack sand to two feet above the top of the well screen. Surging of the well may be required to obtain a good pack around the well screen.
9. Following sand filter pack placement, install a minimum 2-foot thick bentonite seal by slowly adding the pellets to avoid bridging. The bentonite will be hydrated with potable water if the seal is above the water table.
10. Grout the remaining annulus from the top of the bentonite seal to the ground surface using bentonite grout or similar. The grout will be placed into the borehole until the annulus is filled to within three feet of the ground surface.
11. Record the volume of the filter pack, bentonite seal, and grout used.
12. After the grout sets for 24 hours the well completion (flush mount or stick-up) enclosure will be installed. The enclosure will consist of a traffic-rated manhole. Completions will be flush with the surrounding surface. Completions will have a concrete pad sloped slightly away from the manhole. Manholes will have covers secured by bolts.

7.2.6 Surveys

The locations and elevations of any new monitoring wells will be surveyed by a surveyor licensed in the State of New York. At a minimum, the horizontal location of the well will be surveyed to the nearest one foot, the elevation of the ground surface next to the protective casing will be surveyed to the nearest 0.10-foot, and the elevation of the measuring point on the well riser will be surveyed to the nearest 0.01-foot.

7.2.7 Documentation

Observations and data acquired in the field during drilling and installation of monitoring wells will be recorded to provide a permanent record. These observations will be recorded with waterproof ink in a bound weatherproof field logbook and drilling log with consecutively numbered pages. Notes will be recorded daily when in the field. The drilling log is described in detail in SOP No. 1. The information in the field book will include the following as a minimum:

Field Logbook

Project name and number

Observer's name

Visitors and contractors on site

Drilling and well installation observations as described in Section 9.2

Decontamination observations as described in SOP No. 8

Weather conditions

The well installation details will be shown in a diagram which will be drawn in the field book.

Each well diagram will consist of the following (denoted in order of decreasing depth from ground surface):

Bottom of the boring

Casing depth (if intermediate casing is left in the hole)

Screen location(s)

Filter pack intervals

Bentonite seal intervals

Cave-in locations

Height of riser without cap (above ground surface)

Protective casing detail

Additional documentation for well construction in the field book will include the following:

Grout, sand, and bentonite volume calculations prior to well installation

The quantity and composition of the grout, seals, and filter pack actually used during construction

Screen slot size (in inches), slot configuration, outside diameter, nominal inside diameter, schedule/thickness, composition, and manufacturer

Coupling/joint design and composition

Protective casing composition and nominal inside diameter

Start and completion dates

Discussion of all procedures and any problems encountered during drilling and well construction.

7.3 Well Development

The purpose of well development is to remove well drilling fluids, solids, or other particulates which may have been introduced or deposited on the boring wall in a recently installed well during drilling and construction activities. This restores the hydraulic conductivity of the aquifer material surrounding the well to near pre-well installation conditions. Properly developed monitoring wells allow for the collection of groundwater samples that are chemically and physically representative of the aquifer. The procedure is also applicable to older or improperly developed wells which are suspected of not providing representative groundwater samples. This section describes the equipment, methods, and documentation that will be used for developing groundwater monitoring wells.

7.3.1 Equipment and Materials List

The following items are required to properly develop groundwater monitoring wells:

Well keys

Electronic water level indicator (oil/water interface probe for fuel sites)

Calculator

Field notebook

Waterproof pen

Electric inertial pump

Electric submersible pump and controller of appropriate size for the well diameter

Portable electric generator for submersible pumps

Disposable PE bailer (sized appropriately for well)

Nylon or polypropylene rope or wireline (for deep wells) for bailing

Multi-parameter water quality system with a flow-through cell for real-time groundwater parameter monitoring (temperature, pH, specific conductance, DO and ORP), with appropriate calibration solutions

Nephelometric turbidity meter

Polyethylene or glass container (for field parameter measurements)

Plastic spray bottle filled with deionized water

Drums or other large container for development water

Appropriate health and safety equipment

Liquinox® solution

Potable water for decontamination

Distilled or deionized water

Decontamination buckets/pails

Plastic brushes

Well completion information

Well development log

7.3.2 Procedure

The development of a newly installed monitoring well will proceed only after the cement/bentonite grout has been allowed to set for a minimum of 24 hours if such grout was used for constructing a well, and after the completed well has been allowed to equilibrate for at least 48 hours. Monitoring well development activities will be completed prior to purging and groundwater sampling for analytical testing. Before development begins, the development equipment will be decontaminated according to the procedures described in SOP No. 8. Equipment coming in contact with the well will also be decontaminated between wells.

Before development begins, the field personnel will verify that the multi-parameter water quality system, and water level probe are operating properly. The electronic water quality instruments require daily calibration or calibration checks prior to use. Calibration times and readings will be recorded in the field notebook and on calibration forms (SOP No. 11). Specific instructions for calibrating the various water quality instruments are provided in instrument-specific instruction manuals and in SOP No. 11.

Monitoring well development at the former Griffiss AFB will be accomplished by using a bailer, a submersible pump, or an inertial pump to flush the screen, sand pack material, and borehole wall of fine sediment resulting from well drilling and installation activities. This procedure also allows for the removal of fine sediment which may have accumulated within the inner well casing.

Development consists of removing a minimum of five well casing volumes of water during repeated surging and well evacuation episodes. Well surging is the process of causing water to move through the screen and into and out of the sand pack and aquifer formation. This will be accomplished by surging the entire length of well screen using bailer or pump. Surging may also be used during well construction to compact the sand filter pack around the well screen.

Well evacuation is the process of removing water from throughout the entire water column by periodically lowering and raising the pump intake or the point to which the bailer is lowered. Development water will be collected in drums or holding tanks for characterization. The volume of water required for removal during development is calculated using the following method:

1. Measure the depth to water in the well from the measuring point. This is usually the top of the well riser cap which has previously been surveyed.
2. Measure the total depth of the well from the same measuring point used for measuring the depth to water.
3. Calculate the height of water in the well casing by subtracting the depth of water from the total well depth.

4. Calculate the number of gallons of water corresponding to one well volume. This is done by multiplying the height of water column in the well casing by the conversion factor corresponding to the inside diameter of the well casing. The following equation shall be used to calculate the volume of water to be removed during well evacuation:

   For 2-inch well: Well Volume = (Total Well Depth – Water Level Depth X 0.17 gal/ft = gallons/1 well casing volume

   Multiply the volume of one well casing volume by five to obtain the minimum volume of water to be evacuated.

During the well development activities field measurements of temperature, pH, nephelometric turbidity, specific conductance, and dissolved oxygen are made, and the clarity, color, any presence of odors, and other comments regarding water quality are noted in the field notebook and on the well development log. The date, time, and volume of water removed are also recorded at this time. All measurements will be recorded for each well volume of water removed. A sample of water will be collected for measurement of the field water quality parameters at the beginning of well development in order to establish a baseline for comparison with the water quality as well development proceeds.

Initial monitoring well development activities with the bailer or pump will continue until at least five well casing volumes have been removed and measurements of the field parameters have stabilized within 10 percent or 0.1 units and the water removed from the well is as clear of sediment as is practical. Regardless of the clarity of the water removed, a minimum of five well volumes of water will be removed during the bailing/surging phase of well development. If the well is bailed dry, it will be allowed to recover. After initial development activities with the bailer are completed, the well will be further developed by purging after installing the submersible pump and lift pipe. Purging will continue with the submersible pump until the field water quality parameters are within 10 percent or 0.1 units for three consecutive readings.

7.3.3 Documentation

Documentation of observations and data acquired in the field will provide information on well development and also provide a permanent record. These observations and data will be recorded with waterproof ink in a bound weatherproof field book with consecutively numbered pages and on the well development form.

As part of the development process, the following information will be recorded in the field book:

Well designation

Well location

Field personnel

Date(s) and time of well development

Static water level from top of well casing before and after development

Volume of water in well prior to development

Volume of water removed and time of removal

Depth from top of well casing to bottom of well

Screen length

Depth from top of well casing to top of sediment inside well, if present, before and after development

Field measurements of pH, conductivity, turbidity, dissolved oxygen, and temperature taken during and after development

Physical character of removed water throughout development (color, odor, and turbidity)

Type and size/capacity of pump and/or bailer

Description of development technique

Decontamination observations

Instrument calibration record

8 Boring and Monitoring Well Abandonment

8.1 Purpose and Scope

This document defines the SOP for abandoning borings and gives descriptions of equipment and field procedures necessary to abandon borings and monitoring wells at the former Griffiss AFB. This SOP is intended to be used with the UFP QAPP and with other SOPs listed below:

- SOP No. 1: Soil Sampling
- SOP No. 2: Groundwater Sampling
- SOP No. 8: Monitoring Well Installation and Development

8.2 Boring Abandonment Procedures

8.2.1 Equipment and Materials List

The following is an equipment and materials list for boring abandonment:

High solids bentonite grout or granular bentonite

Potable water

Logbook

Boring log sheets

Waterproof and permanent marking pens

Appropriate health and safety equipment

8.2.2 Abandonment Procedures

Borings:
Following completion of the soil borings, each boring must be abandoned and plugged to provide a low-permeability zone that would retard movement of water through the boring backfill. Where water was not encountered and the boring sidewalls are stable the boring may be backfilled using granular bentonite. The dry granular bentonite is poured into the boring from the ground surface filling the boring in 1-foot lifts. Hydration of the bentonite with 1 gallon of water is necessary for each lift.

Monitoring Wells:
All abandonment of monitoring wells, shall be performed in accordance with 6 NYCRR Part 360-2.11 (a)(8)(vi) and the 1996 version Ground-Water Monitoring Well Decommissioning Procedures, Sections 2.2, 9.0, and 10.0. NYSDEC approved abandonment methods into grout and pull, grout in place or over drilling. These are described below:

Grout and Pull
Well casing is pulled out of the ground using a drill rig and a slurry is applied to bore hole.

Grout in place
Well casing remains in the ground; however, a slurry is applied to the well to close all potential pathways.

Over Drilling
Well casing is over drilled by a drilling company. An auger is advanced to the bottom of the well and a slurry is applied to bore hole.

When slurry is used, a mud balance and/or Marsh Funnel shall be used to ensure that the density (lbs/gal) of the abandonment mud mixture conforms to the manufacturer's specification. All abandoned monitoring wells shall be checked 24 to 48 hours after mud/solid bentonite emplacement to determine whether curing is occurring properly. More specific curing specifications or quality assurance checks may be recommended by the manufacturer and shall be followed. Additionally, if significant settling has occurred, a sufficient amount of mud/solid bentonite shall be added to attain its initial level. These slurry/solid bentonite curing checks and any addition of mud/solid bentonite shall be recorded in the field logs.

8.2.3 Pavement Repair

Where borings or monitoring wells penetrate concrete or asphalt pads, it will be necessary to patch the pavement surface following backfilling. Concrete pavements should be filled with low slump (less than 4 inches) concrete mix. Asphaltic or concrete pavements should be filled with asphaltic concrete patch mix and thoroughly compacted by ramming. The surface of any patch should be level upon completion. In freezing weather the concrete mix must be protected from freezing for 48 hours after placement.

8.3 Documentation

Observations and data acquired in the field during boring abandonment will be recorded to provide a permanent record. These observations will be recorded with waterproof black ink in a bound weatherproof field book with consecutively numbered pages. A note shall be placed on the boring log for the boring that was abandoned and backfilled that identifies the date and method of abandonment. The type of material used to patch a pavement surface (if done) will also be noted on the boring log and the field book.

9 Equipment Calibration

9.1 Purpose and Scope

This SOP describes the procedures for equipment calibration and documentation. This SOP is intended to be used with the UFP-QAPP, FSP and with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 8, Monitoring Well Installation and Development

9.2 Equipment and Materials List

The following section provide a list of equipment that may be needed to perform equipment calibration.

Horiba U-22 and Horiba U-52:

Horiba U-22

Horiba U-52

Auto calibration solution pH 4

Calibration cup

Calibration log for Horibas

YSI 556

YSI 556

Calibration cup

Calibration log for YSI

DI water

Conductivity solution (1.413 µS/cm)

pH 4 solution

pH 7 solution

ORP solution (240 mV)

PID, miniRAE

PID, miniRAE

Tedlar bag

Isobutylene (100 ppm)

Calibration log for PID

9.3 Equipment Calibration Procedures

The following provides the procedures for the calibration of the Horiba U-22 and U-52, YSI 556, and PID miniRAE.

Horiba U-22:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins.
- END appears when calibration is complete.
- Press MEAS button and collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-22 manual.

Horiba U-52:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins when the parameters on screen start to blink.
- When parameters stop blinking, calibration is complete.
- Collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-52 manual.

YSI 556:

- Turn on YSI 556.
- Press ESC which will lead to main menu.
- Scroll to Calibrate and press ENT.
- Scroll to DO, press enter, scroll to DO%
- Enter barometric pressure.
- Place probe in DI water (in calibration cup) and loosely tighten probe to calibration cup.
- Press enter, and then enter again.
- DO% is instantly calibrated.
- Acceptable range is 95% to 105%.
- Press ESC to return to calibration menu.
- Scroll to Conductivity, press enter, scroll to Conductivity in list and press enter
- Enter standard, 1.413 µs/cm.

- Fill calibration cup with conductivity solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- Conductivity is instantly calibrated.
- Acceptable range is 1.408 to 1.418 µs/cm.
- Press ESC to return to calibration menu.
- Scroll to pH, press enter, scroll to 2-point calibration and press enter
- Enter 1st standard, 4.00.
- Fill calibration cup with pH 4.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 3.95 to 4.05.
- Press enter.
- Enter 2nd standard, 7.00.
- Fill calibration cup with pH 7.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 6.95 to 7.05.
- Press ESC to return to calibration menu.
- Scroll to ORP, press enter
- Enter standard, 240 mV.
- Fill calibration cup with ORP solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- Conductivity is instantly calibrated.
- Acceptable range is 235 to 245 mV.
- If any errors appear, refer to YSI 556 manual.

PID miniRAE:

Zero Calibration

Turn on PID to Zero Calibration menu.

Press [Y/+] to start calibration.

Press [MODE] to quit and return to the main calibration display.

Zero calibration starts.

When Zero calibration is complete, you see this message: Zeroing is done!, Reading = 0.000 ppm.

Span Calibration

Turn on PID to Scan Calibration menu.

The span gas is first be filled into a Tedlar bag.

Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Press [Y/+] to enter Span calibration.

Turn on your span calibration gas.

Press [Y/+] to initiate calibration.

Span calibration starts and displays this message: Calibrating...

When Span calibration is complete, you see this message: Span 1 is done!, Reading = 100.0 ppm

Per the Mini RAE manual, there is no set range of what is allowed above or below 100 ppm. The Manual simply states that the “reading should be very close to the span gas value”.

9.4 Documentation:

Documentation for equipment calibration forms which are included in Daily CQCRs. The calibration forms include:

Equipment model and number

Date

Calibration personnel

Standard calibration values

Scan gas concentration for PID calibration

Standard calibration solution parameters for water quality

Attachment 1
Field Forms

HTW DRILLING LOG

1. COMPANY NAME	2. DRILLING CONTRACTOR	HOLE NO. SHEET 1 OF 2 SHEETS
3. PROJECT	4. LOCATION	
5. NAME OF DRILLER	6. MANUFACTURER'S DESIGNATION OF DRILL	
7. SIZES & TYPES OF DRILLING & SAMPLING EQUIPMENT	8. HOLE LOCATION	
	9. SURFACE ELEVATION	
	10. DATE STARTED / 11. DATE COMPLETED	
12. OVERBURDEN THICKNESS	15. DEPTH GROUNDWATER ENCOUNTERED	
13. DEPTH DRILLED INTO ROCK	16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED	
14. TOTAL DEPTH OF HOLE	17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES				
20. SAMPLES FOR CHEMICAL ANALYSIS	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21. TOTAL CORE REC %	
22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR			

ELEV. a	DEPTH b	DESCRIPTION OF MATERIALS c	Field Screening Results d	Geotech Sample or Core Box No. e	Analytical Sample No. f	Blow Counts g	REMARKS h

HTW DRILLING LOG

HOLE NO.

PROJECT

INSPECTOR

SHEET 2
OF 2 SHEETS

ELEV. a	DEPTH b	DESCRIPTION OF MATERIALS c	Field Screening Results d	Geotech Sample or Core Box No. e	Analytical Sample No. f	Blow Counts g	REMARKS h

PROJECT

HOLE NO.

WELL PURGING & SAMPLING FORM

Project: _____ Sampled by: _____

Location and Site Code (**SITEID**): _____

Well No. (**LOCID**): _____ Well Diameter (**SDIAM**): _____

Date (**LOGDATE**): _____ Weather: _____

CASING VOLUME INFORMATION:

Casing ID (inch)	1.0	1.5	2.0	2.2	3.0	4.0	4.3	5.0	6.0	7.0	
Unit Casing Volume (A) (gal/ft)	0.04	0.09	0.16	0.2	0.37	0.65	0.75	1.0	1.5	2.0	2.6

PURGING INFORMATION:

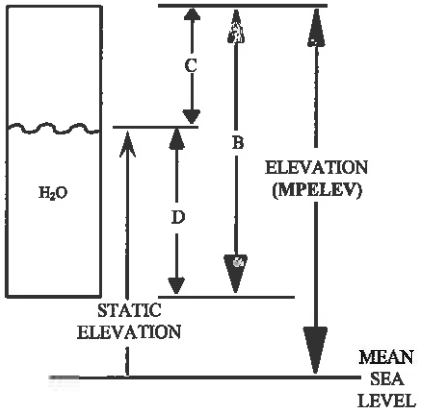
Measured Well Depth (B) (**TOTDEPTH**) _____ ft.

Measured Water Level Depth (C) (**STATDEP**) _____ ft.

Length of Static Water Column (D) = _____ (B) - _____ (C) = _____ (D) ft.

Casing Water Volume (E) = _____ (A) x _____ (D) = _____ gal

Minimum Purge Volume = _____ gal (3 well volumes)



Purge Date and Method: _____

Physical Appearance/Comments: _____

FIELD MEASUREMENTS:

Allowable Range: ± 0.1 ± 5% ±1°C

Time	Volume Removed (gal)	pH	EC (mS/cm)	Temp. (F or C)	Turbidity (NTU)	D.O. (mg/L)	ORP (mV)

Sample Time: _____ Sample ID: _____

Note: Attempt to get at least 5 sets of field measurements during purging. Sample may be collected after 3 to 5 well volumes have been removed and parameters have stabilized. Sample may be collected after 6 well volumes if parameters do not stabilize. VOC and gas sensitive (e.g. alkalinity, Fe^{2+}, CH_4, H_2S) parameters should be sampled first.

WELL PURGING & SAMPLING FORM
(LOW FLOW)

Project: _____ Sampled by: _____

Location and Site Code (**SITEID**): _____

Well No. (**LOCID**): _____ Well Diameter (**SDIAM**): _____

Date (**LOGDATE**): _____ Weather: _____

CASING VOLUME INFORMATION:

Casing ID (inch)	1.0	1.5	2.0	2.2	3.0	4.0	4.3	5.0	6.0	7.0	8.0
Unit Casing Volume (A) (gal/ft)	0.04	0.09	0.16	0.2	0.37	0.65	0.75	1.0	1.5	2.0	2.6

PURGING INFORMATION:

Measured Well Depth (B) (**TOTDEPTH**) _____ ft. (optional)

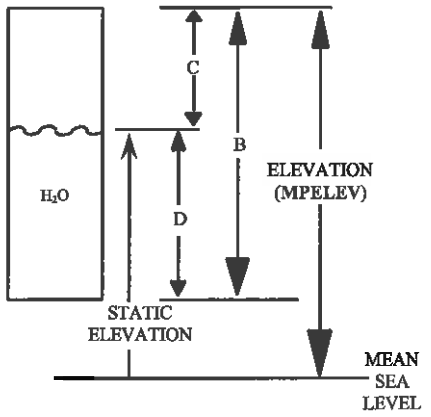
Measured Water Level Depth (C) (**STATDEP**) _____ ft.

Length of Static Water Column (D) = _____ (B) - _____ (C) = _____ (D) ft. (optional)

Pump Intake Depth (ft): _____

Depth during Purging/Sampling: _____ ft
(provide range)

Comments (re: Depth during purging/sampling): _____



Purge Date and Method: BLADDER PUMP _____

Physical Appearance/Comments: _____

Dissolved Ferrous Iron (mg/L): _____

FIELD MEASUREMENTS:

Allowable Range:		± 0.1	± 3%		± 10%	± 10%	± 10mV	
Time	Depth to Water (ft BTOC)	pH	EC (mS/cm)	Temp. (F or C)	Turbidity (NTU)	D.O. (mg/L)	ORP (mV)	Flow Rate (mL/min)

Sample Time: _____ Sample ID: _____

Note: Maintain a flow rate of 200-500 mL/min during purging. Collect samples at a flow rate between 100-250 mL/min. VOC and gas sensitive (e.g. alkalinity, Fe^{2+}, CH_4, H_2S) parameters should be sampled first.

SOIL / SEDIMENT SAMPLING FORM

Project: _____ Sampled by: _____

Site and Site Code (**SITEID**): _____

Sampling Location ID. (**LOCID**): _____

Date (**LOGDATE**): _____ Time: _____

FIELD OBSERVATIONS:

Sample Depth or Interval	**Material Description/ Color**

Comments/Observations:

_____

_____

_____

_____

Sample Time: _____ Sample ID: _____

SOIL VAPOR PROBE MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TRACER GAS VERIFIED: ☐ Yes ☐ No TRACER GAS CONC. (%): _____

SAMPLE PURGE VOLUME: _____

VOLUME OF SOIL VAPOR EXTRACTED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

APPARENT MOISTURE CONTENT: (DRY/MOIST/SATURATED/ETC.)

_____

Comments/Observations during sampling (odor, other instrument readings):

_____

_____

If sampling near an industrial/commercial building, VOCs used during normal operations of facility:

_____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Wind speed/direction: _____

SUB-SLAB VAPOR PROBE MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TRACER GAS VERIFIED: ☐ Yes ☐ No TRACER GAS CONC. (%): _____

SAMPLE PURGE VOLUME: _____

VOLUME OF SOIL VAPOR EXTRACTED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

APPARENT MOISTURE CONTENT: (DRY/MOIST/SATURATED/ETC.)

_____

Comments/Observations during sampling (spills, floor stains, odors, other instrument readings):

VOCs used during normal operations of facility:

_____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Precipitation: _____

Ventilation conditions: _____

Heating System Active? ☐ Yes ☐ No Indoor Air Temp: _____

Location in relation to sample location: _____

Windows Closed? ☐ Yes ☐ No

INDOOR/OUTDOOR AIR MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TYPE OF SAMPLE: ☐ INDOOR ☐ OUTDOOR

DURATION OF AIR SAMPLING: _____

VOLUME OF AIR SAMPLED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

Comments/Observations during sampling (spills, floor stains, odors, other instrument readings):

_____

_____

VOCs used during normal operations of facility: _____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Precipitation: _____

Ventilation conditions: _____

Heating System Active? ☐ Yes ☐ No Indoor Air Temp.:_____

Location in relation to sample location: _____

Windows Closed? ☐ Yes ☐ No

WEATHER OBSERVATION FORM

LOCATION: _____

DATE: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

Thermometer: _____

Anemometer: _____

	Time (military)	Precip. (in)	Atmospheric pressure (in)	Temp. (degrees F)	Wind (mph)	Comments
Prior to Sampling						
Mid Day						
End of Sampling						

Notes: Additional measurements should be taken in case of weather condition changes.
Air sampling will be postponed if conditions move outside the acceptable range.

Sampling Event Acceptable Range:
1. Precipitation: dry while conducting sampling.
2. Atmospheric pressure: 29.7 – 30.4 in Hg.
3. Temperature: 35 – 95 degrees F. The ground must be completely thawed.
4. Wind: <10 mph.

NEW YORK STATE DEPARTMENT OF HEALTH
INDOOR AIR QUALITY QUESTIONNAIRE AND BUILDING INVENTORY
CENTER FOR ENVIRONMENTAL HEALTH

This form must be completed for each residence involved in indoor air testing.

Preparer's Name _____ Date/Time Prepared _____

Preparer's Affiliation _____ Phone No._____

Purpose of Investigation_____

1. OCCUPANT:

Interviewed: Y / N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

Number of Occupants/persons at this location _____ Age of Occupants _____

2. OWNER OR LANDLORD: (Check if same as occupant ___)

Interviewed: Y / N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

3. BUILDING CHARACTERISTICS

Type of Building: (Circle appropriate response)

Residential	School	Commercial/Multi-use
Industrial	Church	Other: _____

If the property is residential, type? (Circle appropriate response)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouses/Condos
Modular	Log Home	Other:_____

If multiple units, how many? _____

If the property is commercial, type?

Business Type(s) _____

Does it include residences (i.e., multi-use)? Y / N If yes, how many? _____

Other characteristics:

Number of floors_____ Building age_____

Is the building insulated? Y / N How air tight? Tight / Average / Not Tight

4. AIRFLOW

Use air current tubes or tracer smoke to evaluate airflow patterns and qualitatively describe:

Airflow between floors

_____

_____

_____

Airflow near source

_____

_____

_____

Outdoor air infiltration

_____

_____

_____

Infiltration into air ducts

_____

_____

_____

5. BASEMENT AND CONSTRUCTION CHARACTERISTICS (Circle all that apply)

a. Above grade construction:	wood frame	concrete	stone	brick
b. Basement type:	full	crawlspace	slab	other _____
c. Basement floor:	concrete	dirt	stone	other _____
d. Basement floor:	uncovered	covered	covered with _____	
e. Concrete floor:	unsealed	sealed	sealed with _____	
f. Foundation walls:	poured	block	stone	other _____
g. Foundation walls:	unsealed	sealed	sealed with _____	
h. The basement is:	wet	damp	dry	moldy
i. The basement is:	finished	unfinished	partially finished	
j. Sump present?	Y / N			
k. Water in sump?	Y / N / not applicable			

Basement/Lowest level depth below grade: _____(feet)

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, drains)

_____

_____

6. HEATING, VENTING and AIR CONDITIONING (Circle all that apply)

Type of heating system(s) used in this building: (circle all that apply – note primary)

Hot air circulation	Heat pump	Hot water baseboard	
Space Heaters	Stream radiation	Radiant floor	
Electric baseboard	Wood stove	Outdoor wood boiler	Other _____

The primary type of fuel used is:

Natural Gas	Fuel Oil	Kerosene
Electric	Propane	Solar
Wood	Coal	

Domestic hot water tank fueled by: _____

Boiler/furnace located in:	Basement	Outdoors	Main Floor	Other_____
Air conditioning:	Central Air	Window units	Open Windows	None

Are there air distribution ducts present? Y / N

Describe the supply and cold air return ductwork, and its condition where visible, including whether there is a cold air return and the tightness of duct joints. Indicate the locations on the floor plan diagram.

_____

_____

_____

_____

7. OCCUPANCY

Is basement/lowest level occupied? Full-time Occasionally Seldom Almost Never

Level	**General Use of Each Floor (e.g., familyroom, bedroom, laundry, workshop, storage)**
Basement	_____
1st Floor	_____
2nd Floor	_____
3rd Floor	_____
4th Floor	_____

8. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

a. Is there an attached garage? Y / N

b. Does the garage have a separate heating unit? Y / N / NA

c. Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, atv, car) Y / N / NA Please specify_____

d. Has the building ever had a fire? Y / N When?_____

e. Is a kerosene or unvented gas space heater present? Y / N Where?_____

f. Is there a workshop or hobby/craft area? Y / N Where & Type? _____

g. Is there smoking in the building? Y / N How frequently? _____

h. Have cleaning products been used recently? Y / N When & Type? _____

i. Have cosmetic products been used recently? Y / N When & Type? _____

j. Has painting/staining been done in the last 6 months? Y / N Where & When? _____

k. Is there new carpet, drapes or other textiles? Y / N Where & When? _____

l. Have air fresheners been used recently? Y / N When & Type? _____

m. Is there a kitchen exhaust fan? Y / N If yes, where vented? _____

n. Is there a bathroom exhaust fan? Y / N If yes, where vented? _____

o. Is there a clothes dryer? Y / N If yes, is it vented outside? Y / N

p. Has there been a pesticide application? Y / N When & Type? _____

Are there odors in the building? Y / N
If yes, please describe: _____

Do any of the building occupants use solvents at work? Y / N
(e.g., chemical manufacturing or laboratory, auto mechanic or auto body shop, painting, fuel oil delivery, boiler mechanic, pesticide application, cosmetologist

If yes, what types of solvents are used? _____

If yes, are their clothes washed at work? Y / N

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, use dry-cleaning regularly (weekly) No
Yes, use dry-cleaning infrequently (monthly or less) Unknown
Yes, work at a dry-cleaning service

Is there a radon mitigation system for the building/structure? Y / N Date of Installation: _____
Is the system active or passive? Active/Passive

9. WATER AND SEWAGE

Water Supply:	Public Water	Drilled Well	Driven Well	Dug Well	Other: _____
Sewage Disposal:	Public Sewer	Septic Tank	Leach Field	Dry Well	Other: _____

10. RELOCATION INFORMATION (for oil spill residential emergency)

a. Provide reasons why relocation is recommended: _____

b. Residents choose to: remain in home relocate to friends/family relocate to hotel/motel

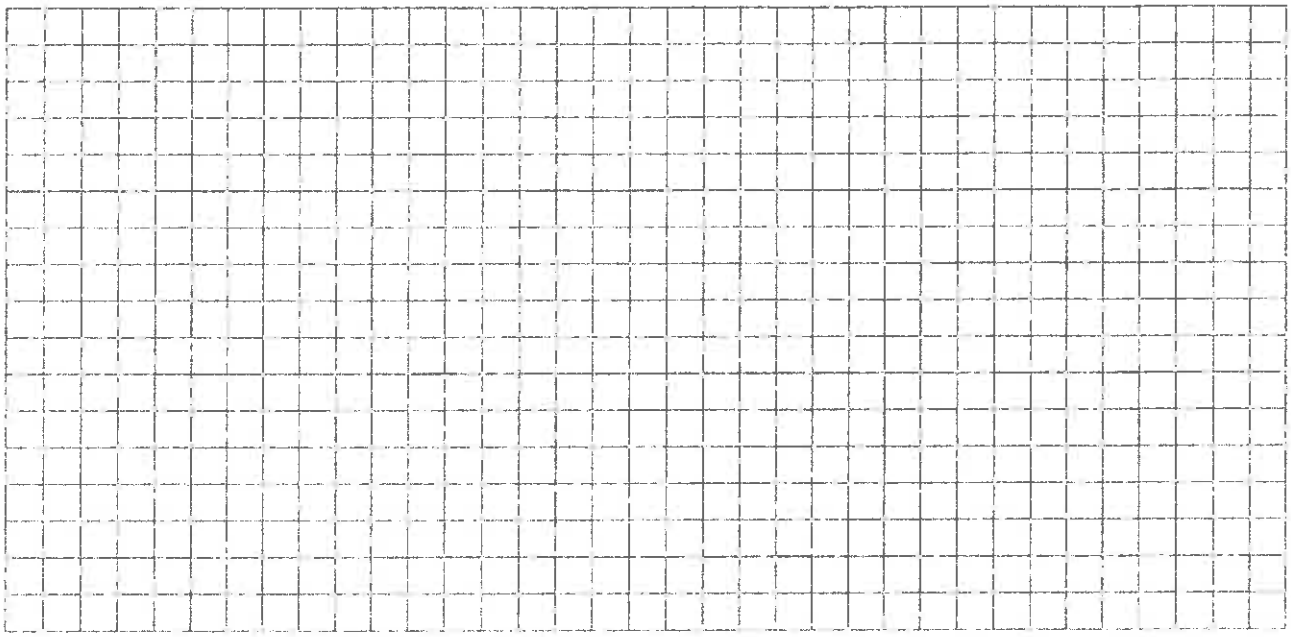
c. Responsibility for costs associated with reimbursement explained? Y / N

d. Relocation package provided and explained to residents? Y / N

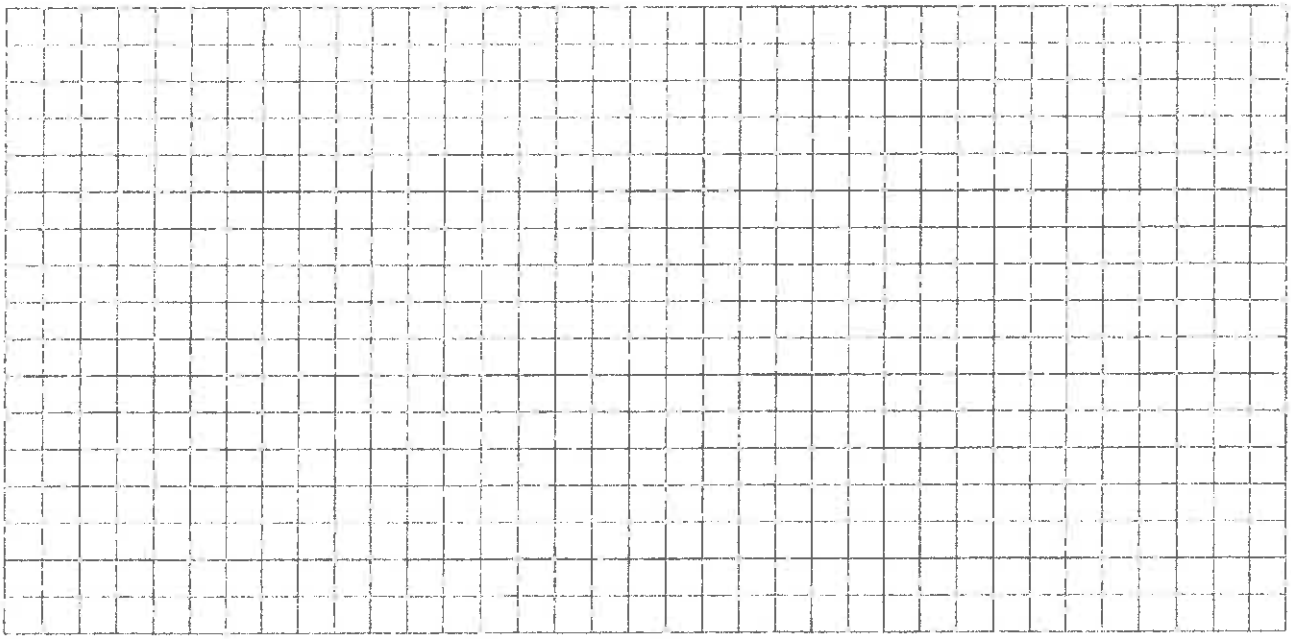
11. FLOOR PLANS

Draw a plan view sketch of the basement and first floor of the building. Indicate air sampling locations, possible indoor air pollution sources and PID meter readings. If the building does not have a basement, please note.

Basement:



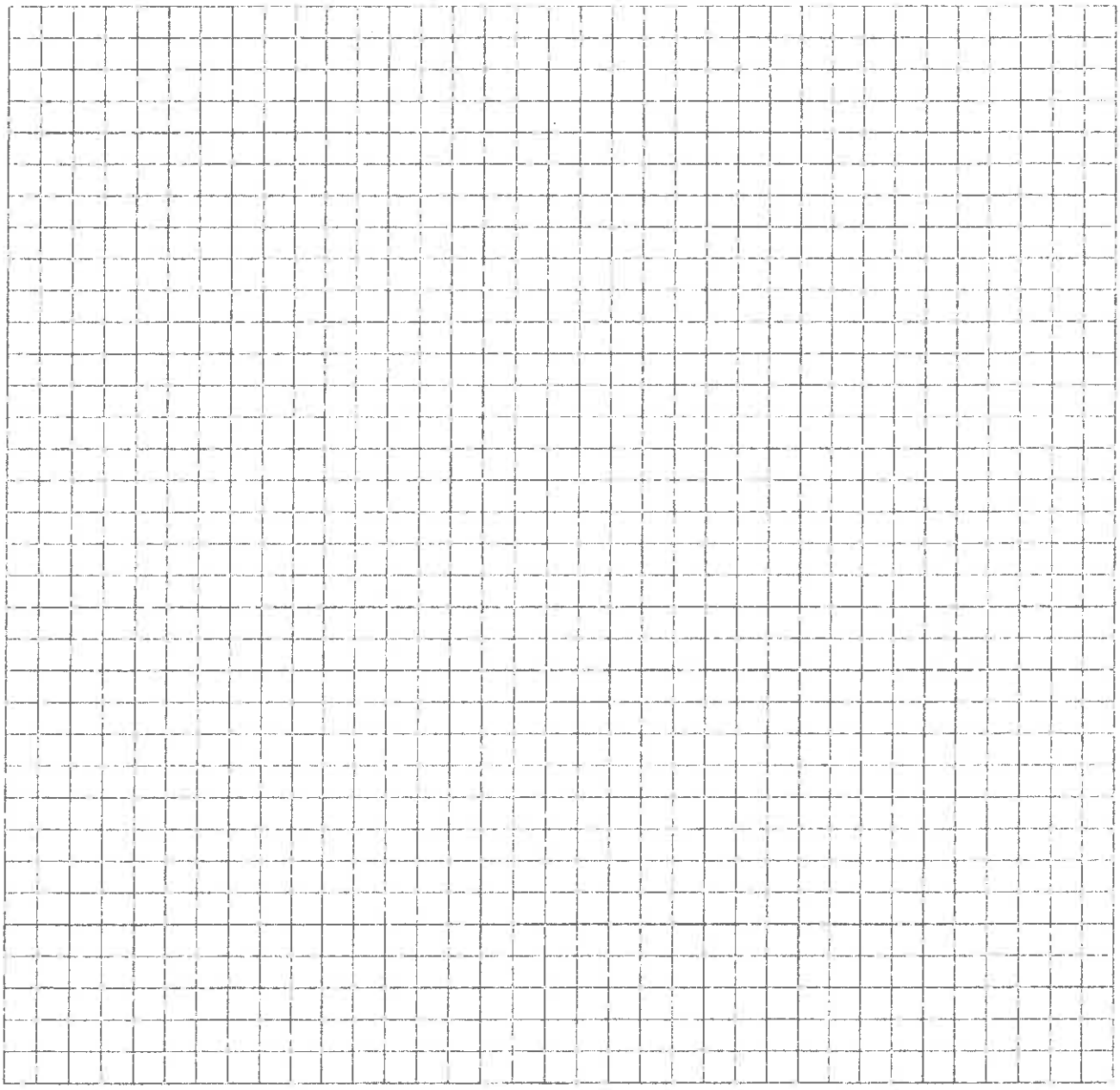
First Floor:



12. OUTDOOR PLOT

Draw a sketch of the area surrounding the building being sampled. If applicable, provide information on spill locations, potential air contamination sources (industries, gas stations, repair shops, landfills, etc.), outdoor air sampling location(s) and PID meter readings.

Also indicate compass direction, wind direction and speed during sampling, the locations of the well and septic system, if applicable, and a qualifying statement to help locate the site on a topographic map.



13. PRODUCT INVENTORY FORM

Make & Model of field instrument used: _____

List specific products found in the residence that have the potential to affect indoor air quality.

Location	Product Description	Size (units)	Condition*	Chemical Ingredients	Field Instrument Reading (units)	Photo ** Y / N

* Describe the condition of the product containers as **Unopened (UO), Used (U),** or **Deteriorated (D)**
** Photographs of the **front and back** of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

Daily Health and Safety Meeting Form

Date: _____ *Time :* _____

Location: FPM office (sample room)

Weather Conditions: _____

Meeting Type: Daily Health and Safety

Personnel Present:

_____

_____

Visitors Present: _____

Visitor Training: _____

PPE Required: Modified D

Possible risks, injuries, concerns:

_____

_____

_____

Anticipated Releases to Environment (if so, describe and detail response action/control measures implemented):

_____

_____

Property Damage:

_____

_____

Description (include sequence of events describing step by step how incident happened):

_____

_____

Analysis for, and Implementation of Corrective/Preventative Procedure to Prevent Future Occurrences (to be formulated by SSHO + FOM, approved by PM, and SSHO implemented):

_____

_____

_____

Report made by (Name): _____

SSHP Organization Title: Site Safety and Health Officer

Daily Health and Safety Inspection Form

Date:_____ *Time:* _____

Location: _____

Personnel Present: _____

_____

_____

*Visitors Present:*_____

_____

Behavior, approach or practice that was found unacceptable:

_____

_____

_____

*Possible risks, injuries, concerns, deviations from H&S Plan:*_____

_____

_____

_____

Anticipated releases to environment or anticipated future Health and Safety risks: _____

_____

_____

*Analysis for, and Implementation of Corrective/Preventative Procedure to Prevent Future Occurrences (to be formulated by SSHO and FOM, approved by PM, and implemented by SSHO):*_____

_____

_____

_____

_____

Report made by (Name): _____

SSHP Organization Title: ____Site Safety and Health Officer_____

_____

_____

_____

Well Decommissioning Form

Date	Well ID	Depth to Water (TOIC)	Total Depth (TOIC)	Method of Decommissioning	Physical Description (Well Type, etc.)	Grout Added (gallons)

Appendix B

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

Title: Determination of VOCs in Ambient Air EPA Compendium Methods TO14 and TO15

Approval Signatures:

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Approval Date: August 16, 2012

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the analysis of polar and non-polar volatile organic compounds (VOCs) in ambient air. The procedure is applicable to those VOCs that have been evaluated by the laboratory for their consistent performance in meeting the control criteria put forth in Compendium Method TO-15. While the compendium method is specifically written for the analysis of samples collected in leak-free passivated stainless steel canisters, it may be applied to the analysis of samples that have employed the use of other collection devices such as Tedlar bags and solid absorbents.

1.1 Analytes, Matrix(s), and Reporting Limits

The target compound list and reporting limits for each compound are provided in Table 1.

2.0 Summary of Method

An aliquot of sample is pulled from the canister through a solid multi sorbent bed trap which reduces the water content of the sample. The sample is thermally desorbed and the VOCs are carried onto a GC column coupled to a mass spectrometer. Compounds are identified by comparison of the mass spectra for individual peaks in the total ion chromatogram to the fragmentation patterns of ions corresponding to VOCs including the intensity of primary and secondary ions as well as the patterns of stored spectra acquired under similar conditions. The concentration of the target compound is calculated by internal standard technique using the average response factor of that compound as determined by the initial calibration.

This procedure is based on EPA Compendium Method TO-15 "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.

If the laboratory has modified the method, a list of these modifications may be found in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Contamination may occur if canisters or other equipment is not properly cleaned before use. The laboratory procedures for canister and flow controller cleaning procedures are provided in Appendices C and D.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples

and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The analytical system contains zones with elevated or depressed temperatures that are capable of causing injury upon direct contact. The analyst needs to be aware of the locations of those zones, and allow them to return to room temperature prior to maintenance activities or take measures to avoid contact with hot and/or cold surfaces. There are areas of high voltage in the analytical system. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Liquid nitrogen (LN2) is used for cryogenic purposes. In addition to avoiding contact with LN2 cooled surfaces, analysts must be aware of the potential for oxygen depletion in a confined space in the event of an unexpected large release of the product. Users should evacuate a confined space in which large amounts of LN2 have been released.

Sample canisters are occasionally pressurized for cleaning or sample dilution purposes. Lab systems are designed to ensure that the cans are not pressurized above 40 psi. Eye protection must be worn when cans are pressurized in the event of a canister failure.

5.2 Primary Materials Used

There are no 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.** 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.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment meets the specification of this SOP.

6.1 Sampling Equipment

- 6L, 3L, and 1L SUMMA® Canisters: Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 Valves, or equivalent.

- 6L SUMMA® Canisters: Silicon lined-Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 valves or equivalent.

- Flow Controllers: Restek Catalog #24239 or equivalent.

- Flow Controller Orifice: Various sizes ranging from 0.008” to 0.060”, Restek or equivalent.

- Flow Controller Vacuum Gauges: Capable of measuring vacuum to an absolute vacuum of -30” of HG, and pressure up to 30 psi, Grainger Catalog #5WZ37 or equivalent.

- Rain Guard: Stainless Steel Tubing ¼", 10ft. Grainger or Equivalent. Cut 8" and bend into a J shape using a pipe bender.

- Stainless Steel Pre-Filter (7 um): Swagelok Catalog# SS-4F-T7-7 or equivalent.

- Teflon Tape: Home Depot Brand or equivalent.

6.2 Analytical System

- Mass Spectrometer: Agilent 5973 or 5972 MSD or equivalent.
- Gas Chromatograph: Agilent 6890 or equivalent.
- VOC Autosampler: Entech 7016CA or equivalent.
- Cryogenic Concentrator: Equipped with an electronic mass flow controller that maintains a constant flow for carrier gas and sample over a range of 0-200 cc/min. Entech 7100A or equivalent.
- Low Pressure Liquid Nitrogen: Air Gas or equivalent.
- Glass Bead Cryotrap: Capable of effectively removing water while trapping polar and non-polar compounds. Entech catalog# 01-04-11320.
- TENAX Sorbent Trap: Capable of removing CO2 and trapping the polar and non-polar compounds. Entech catalog # 01-04-11330.Primary Column: Fused silica capillary column (60 m x 0.32 mm x 1.8 μm), Restek RTX-624 or equivalent.
- Data System: PC software for Entech instrumentation. Hewlett-Packard ChemStation data acquisition software and Hewlett-Packard ChemServer, Target 3.5 data processing software or TestAmerica Chrom and TestAmerica LIMS (TALS).

6.3 Cleaning System

- Canister Cleaner Module and Software: Capable of filling canisters with humidified air and evacuating canisters to 50 mtorr, Entech Model 3100A or equivalent.

- Vacuum Pump: Capable of evacuating sample canisters to full vacuum. Vacuubrand or equivalent.

- Cleaning Manifold: Equipped with stainless steel and Teflon transfer lines and connections for cleaning up to twelve canisters simultaneously.

- Heating Belts: Individual thermal-stated heating belts used to heat canisters to 100ºC during the manifolds cleaning cycles. Entech or equivalent.

- Cleaning oven: Capable of cleaning 6 Summa Cans simultaneously at a temperature of 100ºC. Entech or equivalent.

- Flow Controller Cleaning Manifold: Capable of flushing hot Nitrogen through 24 flow controllers simultaneously for cleaning.

6.4 Miscellaneous Supplies

- Mass Flow Controller, NIST Traceable: Capable of flow rate of 70 mL/min, McMillan Company 80SD or equivalent. Use for the preparation of calibration and working standards.

- Zero Air Generator: Ballston Model HPZA-3000 or equivalent.
- Syringes: Gas tight with a Luer-Lok tip, assorted sizes ranging from 1.0 mL to 1.0 L, SGE or equivalent.

- Digital Pressure Gauges, NIST Traceable: Capable of measuring pressure in the range of -30" Hg to 100 psi, Dwyer Models DPGA-12 and 67100 or equivalent.

- Digital Flow Meter, NIST Traceable: Alltech or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Ultra Pure Humidified Zero Air - Pass ambient laboratory air through a zero air generator. The zero air generator humidifies the air to a relative humidity of >20 percent.

7.2 Standards

Purchase the following stock standard mixtures from commercial vendors:

- Mixed Gas Stock Standard: - Commercially prepared standard that includes internal standard and tune standard compounds: Bromofluorobenzene, Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5, and at a concentration of 100 ppbv each. Spectra Gas or equivalent.

- Calibration Stock Standard: - Commercially prepared custom gaseous stock standard used by all network facilities that includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases or equivalent.

- Calibration Ethanol Neat Material. >99.5 %

- ICV / LCS Stock Standard: - Custom made gaseous stock standard prepared from a different lot(s) of the source material(s) used to manufacture the calibration stock standard. The ICV/LCS stock standard includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases.

- ICV/LCS Ethanol Neat material. >99.5% from a source other than the calibration source.

Prepare calibration and working standards mixtures by diluting a known volume of the stock standard in humidified ultra pure zero air to a specified volume. The formulations for standard preparation are provided in Appendix B along with recommended expiration dates and storage conditions.

Each stock standard is assigned a 1 year expiration date from manufacture and recertified annually. The ethanol neat material is assigned the expiration date given by the manufacturer.

The recertification procedure is as follows:

Internal Standard Mixed Gas Stock: Assay the active internal standard cylinders against a new, vendor certified cylinder purchased from Spectra Gas. Verify that the % difference between new, vendor certified cylinder and the active cylinder is within 20%. If this criterion is not met, replace the cylinder.

Calibration Stock Standard and the ICV/LCS Stock Standard: Return one of the cylinders to Spectra for recertification (rotate a different cylinder each year). When the re-certified stock standard cylinder is returned, assay against all active cylinders. The difference between the recertified value and the assay values should be within 15%. If these criteria are not met, return the cylinder that was not re-certified to the vendor for recertification or purchase a new stock standard.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Air	1L ,3L or 6L Passivated Summa Canister	1L	NA	30 days from collection	EPA TO-15

All samples should be collected in passivated stainless steel canisters that have been certified clean prior to sampling. The laboratory will provide certified clean canisters to the client upon request. The procedures for clean canister certification are provided in Laboratory SOP BR-AT-011.

The laboratory can also provide flow controllers set to the appropriate flow rate for the sampling time required by the client.

The laboratory ships air canisters in custom made boxes. The boxes are equipped with custom-made foam inserts to hold the pre-set flow-controllers. The custom shipping materials are designed to prevent damage of equipment to and from the sampling site. The laboratory checks the equipment to ensure it is in proper working order before shipment to the client and additional checks are performed on return of the equipment to the laboratory. Sampling instructions are provided with each sampling kit. The sampling crew is advised to handle the sampling equipment using the instructions provided by the laboratory to ensure optimum performance.

Samples should be stored at ambient temperature.

The analytical holding time is 30 days from the date of sample collection.

9.0 Quality Control

9.1 Sample QC

The following quality control samples are prepared with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Internal Standard (ISTD)	Every Sample	See Table 3
Laboratory Control Sample Duplicate (LCSD)	Client request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3
Trip Blank (TB)	Client Request	See Table 3

NOTE: The compendium reference method does not require the analysis of a laboratory control sample (LCS) or provide criteria for the evaluation of an LCS. The laboratory performs an LCS at the above mentioned frequency as an evaluation of percent recovery in a blank matrix. The control limits set by the laboratory for the LCS (70-130) are those specified in Section 11.4 of the reference method for the audit accuracy evaluation.

The compendium method does not require analysis of a LCSD. Evaluations for precision should be derived from field samples. Duplicate precision should be measured by the analysis of a sample duplicate. Replicate precision should be measured from separate aliquots taken from the same sample canister. The laboratory will perform an LCSD to measure precision only per client request and analysis of the LCSD is considered a billable. The acceptance criteria for duplicate and replicate precision is <25%. Unless otherwise specified by the client during project initiation, the LCSD will be used to measure precision only. The LCS will be used for evaluations for percent recovery and to determine if corrective action is necessary.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Tune Standard (BFB)	Each Analytical Window	See Section 10.0
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Section 10.0
Initial Calibration Verification (ICV)	Once, after each ICAL	See Section 10.0
RT Window Establishment	Once per ICAL	See Section 10.0
Relative Retention Time (RRT)	With each sample	See Section 10.0
Continuing Calibration Verification (CCV)	Daily, after each BFB	See Section 10.0

10.0 Procedure

10.1 Support Equipment Calibration

Verify the calibration of the mass flow controller used to prepare standards, the calibration of the digital flow meter used to set and check the flow rates of the FC(s) used for sample collection, and the calibration of the digital pressure gauges used to check return canister pressure is current to the year. Immediately notify the QA department if the calibration is not current and

wait for further instruction. Equipment whose calibration has expired may not be used without documented approval from the QA department.

NOTE: The QA department schedules the annual calibrations of the support equipment and maintains all Certificates of Calibration. The flow controllers are checked against a NIST traceable standard. This check is performed by the manufacturer of the equipment, when possible, or by an approved vendor that provides certification service.

10.2 Instrument Calibration

10.2.1 Tune Standard

Analyze a tune standard (BFB) prior at the beginning of each analytical window. The tune standard is a commercially prepared mixed gas stock standard that includes bromofluorobenzene (BFB) at a concentration of 100 ppbv.

To analyze the tune standard:

1) Establish the instrument operating conditions specified in Section 10.4.1.

2) Attach the mixed gas stock standard to the Entech concentrator by attaching the cylinder to the line dedicated for introduction of the internal standard (ISTD). The concentrator directly injects 20 mL of the 100 ppbv stock standard onto the instrument to yield an on column concentration of 10 ppbv.

3) Acquire the data and evaluate the results against the acceptance criteria given in Table 2. Criteria must be met prior to further analysis. The official start time of the 24 hour analytical window is the injection time of a passing tune standard. All samples must be injected within 24 hours of that time.

NOTE: The data processing system averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of BFB.

10.2.2 Initial Calibration (ICAL)

The instrument must be calibrated with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method.

The laboratory routinely analyzes 8 standards at the recommended concentrations of 0.04, 0.20, 0.50, 5.0, 10.0, 15.0, 20 and 40 ppbv, except for Ethanol. For Ethanol, a 6 point curve is analyzed at the following concentrations: 5, 10, 15, 20, 40, and 100 ppbv. Even though seven calibration standards are routinely analyzed not every calibration standard is used for each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five standards. The analyte group associations for each target analyte are provided in Table 1. The calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven point calibration curve. The calibration range is 0.20 to 40 ppbv with the 0.04 ppbv standard routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.20 ppbv

- Group B: This analyte group is associated with a six point calibration curve. The calibration range is 0.50 to 40 ppbv with the 0.04 and 0.20ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.50 ppbv.

- Group C: This analyte group is associated with a five point calibration curve. The calibration range is 5.0 to 40 ppbv with the 0.20, 0.50, and 0.04 ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 5.0 ppbv.

- Group D: This analyte group is an eight point calibration curve. The calibration range is 0.04 40 ppbv. The limit of quantitation (LOQ) for this group of analytes is 0.04 ppbv.

- Group E: (Ethanol : This analyte has a six point calibration curve. The calibration range is 5 to 100 ppbv. The limit of quantitation (LOQ) for this analyte is 5 ppbv.

Prepare the calibration standards using the formulations provided in Appendix B.

Analyze the standards in a sequence from lowest to highest concentration using the instructions provided in Section 10.4.2.

The data processing system calculates a relative response factor (RRF), for each analyte and isomer pair using the assigned internal standard. The internal standard associations for each target analyte are provided in Table 1. The data processing system also calculates a mean relative response factor, relative standard deviation (RSD), relative retention time (RRT) and the mean RRT.

The following criteria must be met for a calibration to be considered acceptable:

- The RSD for each target analyte must be <30% with at most 2 exceptions up to a limit of 40%**.**

- The area response for the primary quantitation ion for the internal standard for each ICAL standard must be within 40% of the mean area response over the calibration range for each internal standard.

- The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

If these criteria are not met inspect the system for problems and perform corrective action. Recommended corrective actions are provided in Section 10.2.5 and in Table 3.

Repeat initial calibration whenever instrument operating conditions are changed, a new column is installed, when significant instrument maintenance has been performed, and when the result of the CCV indicate the calibration is no longer valid.

10.2.3 Second Source Calibration Verification (ICV)

Immediately following an acceptable initial calibration verify the accuracy of the calibration by the analysis of the second source calibration verification standard (ICV).

Prepare the ICV following the formulation provided in Appendix B.

Analyze the ICV following the instructions provided in Section 10.4.2.

The percent recovery (%R) for each target analyte must be within 70-130%. If criteria are not met, perform corrective action. Recommended corrective actions are provided in Table 3. If corrective action is not successful, remake your standards and recalibrate.

If after successful analysis of the ICV, time remains in the 24-hour analytical window, QC and field samples may be analyzed without analysis of a continuing calibration verification check standard. If time does not remain in the analytical window, a new analytical sequence must be initiated with a Tune Standard followed by daily calibration (CCV).

10.2.4 Continuing Calibration Verification (CCV)

Analyze the CCV immediately after the tune standard unless the analytical window includes ICAL, in which case, a CCV is not required.

Prepare the CCV standard using the formulation given in Appendix B. The recommended concentration of the CCV for each target analyte is 10.0 ppbv.

Analyze the CCV following the instructions provided in Section 10.4.2. The data system calculates a response factor for each analyte and calculates the percent difference (%D) of the RRF relative to the mean RRF in the most recent initial calibration.

- The %D for each target analyte must be within ±30%. If the above criteria are not met, repeat the analysis of the CCV once. If the second CCV meets criteria, continue with the analytical sequence. If it fails, evaluate the data to determine if one of the following conditions is met. If these conditions are not met corrective action must be taken. Guidance for troubleshooting is provided in Section 10.2.5. After corrective action the analytical sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If these two CCVs do not meet the criteria, recalibration is required prior to further analysis.

  - If the CCV criteria are exceeded high, indicating a high bias, and the associated samples have non-detects for those analytes, the analytical data may be considered usable. In the absence of instructions otherwise, proceed with analysis.

  - If the CCV criteria are exceeded low, indicating a low bias, analytical results may be reported if those results exceed the project's regulatory decision level. In other words, if the analytical results are sufficiently high to counter the low bias, results may be reported. Consult with the project manager to determine if the exception is allowable for each project.

10.2.5 Troubleshooting

Check the following items in case of calibration failures:

- Loss of sensitivity or unstable ISTD recoveries are usually the result of a leak. Check the union between the GC column and Entech transfer line.

- Loss of sensitivity for individual compounds may be a result of either an active site in a transfer line or a bad trap. Troubleshoot and perform maintenance as necessary.

- Poor chromatography usually requires GC column maintenance, perform as necessary.

- Carryover is usually caused by excessive amounts of analyte introduced to the system. Analyze blanks until the system is cleaned or replace the traps and transfer lines if necessary.

Refer to corporate policy CA-Q-S-005 for additional information of procedures to establish and troubleshoot initial calibration curves.

10.3 Sample Preparation

10.3.1 Post Sampling Canister Pressure Check Procedure

Perform the post-sampling canister pressure check within 1 business day of receipt of canisters in the laboratory so that any problems found are quickly identified and communicated to the client. Record the date and time the post-receipt check is performed is in the analytical record.

To perform the post- sampling canister pressure check:

1) Inspect the condition of equipment for signs of damage. If damage is observed, immediately notify the PM and await further instruction.

2) Record your checks on the Air Canister Post-Sampling Pressure Check Record.

3) Check to see if the sampling FC(s) were returned with the canister(s). If so, check the paperwork (Canister ID Tag, Field Test Data Sheet or Chain of Custody (COC) to determine if the sampling record identifies which FC was used for each canister. If the paperwork does not include this information, record the omission on the post-sampling check record.

   NOTE: The laboratory's sample acceptance policy for air samples in canisters requires that the sampling crew record the ID of the FC used for sample collection on the tag attached to each canister, but the association may also be recorded on the Field Test Data Sheet or a COC. With this information the laboratory can review the history of use of the FC as needed to troubleshoot potential equipment problems. Without the association, the history of use of the FC is unknown. The laboratory strongly recommends that field samplers be instructed to provide this information for each sampling event.

4) Check the pressure of each canister using a digital pressure gauge. Verify that the calibration of the gauge has not expired prior to use. If the calibration sticker indicates calibration checks are past due, notify department management and remove the gauge from service.

Attach the gauge to the canister inlet. Check for the presence of burr or thread damage when attaching the pressure gauge to the canister inlet. If damage is observed, record the observation on the post-sampling check record

Open the canister valve and record the pressure reading.

The pressure should be between -10" Hg to -1"Hg, except for "grab" samples and samples whose sampling time is <200 mL/min, which do not have a return pressure criteria range.

NOTE: The return canister pressure criteria of -10"Hg to -1"Hg was established based on the recommendations provided in Section V of Appendix I of the Vapor Intrusion Guidance Document prepared by the New Jersey Department of Environmental Protection (NJDEP). These sections of this document describe canister and quality assurance requirements for USEPA Methods TO-15 and TO-17. This document explains that due to recent advances in technology in concentrator units, such as with the Entech concentrators used by the laboratory, it is now possible to remove sample from canisters with a negative pressure of -10"Hg without having to add makeup air. Previous models of concentrators (such as NuTechs) required a pressure of at least -5"Hg. When the return negative pressure of a canister is greater than -10"Hg, the laboratory may need to add makeup air to the canister in order to provide a sufficient amount of sample for analysis. The need for the addition of makeup air depends on the concentrator unit. Some concentrator units used by the laboratory can pull sample without the addition of make-up air when the return negative pressure is up to -15"Hg. The amount of air added to the canister depends on the return pressure reading and will vary with each canister. See section 10.3 for the procedure of the determination of amount of makeup air needed. Except for "grab" samples and samples whose sampling time is set for <200 mL/min, the return pressure of a canister should never be zero negative pressure or a pressure equal to ambient pressure. If it is, the PM must consult with the client and obtain authorization for the laboratory to proceed with analysis.

5) If the return pressure is within range for all samples, photocopy the post-sampling canister pressure check record and attach the record to the screen worksheet.

   If the return pressure is not within range, initiate an anomaly report and perform one of the following actions:

   - Action 1: If the FC was not returned with the canister, attach a copy of the post-sampling pressure check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheet and forward the original anomaly report to the QA department.

   - Action 2: If the FC was with the canister, perform a leak check on the FC gauge and re-check the FC's flow rate as follows. Record all measurements on the original Flow Controller Set Flow Rate & Leak Check Record. The protective sticker on the back of the FC will provide the page number that corresponds to the logbook record.

   To check the flow rate and check the FC gauge for leaks:

1) Remove the stem from the FC.

2) Attach a dust cap where the stem was, and attach the FC to the control gauge/vacuum manifold.

3) Turn the calibrated digital flow meter on and zero the meter.

4) Turn on the vacuum pump and read the vacuum of the control gauge and the FC gauge and record these readings.

5) Turn off the vacuum. Wait ~30 seconds and read the vacuum of the control gauge and the FC gauge and record these readings.

6) The difference between the initial and final readings for the control gauge should be zero. If it is not, there is a leak in the manifold system. Stop work and correct the problem.

7) The difference between the initial and final readings for the FC gauge should be zero. If it is not, there is a leak in the FC assembly. Record the presence of a leak in the record and set aside the FC for service and repair.

8) Remove the dust cap from the FC and re-attach the stem.

9) Attach the flow meter tube to the stem of the FC.

10) Measure the flow rate. The flow rate should be within the ranges specified in the Set Flow Rate Table (See Table 4) for the sampling time requested. If the flow rate is not within range, record the situation on the anomaly report.

11) Attach a photocopy of the Flow Controller Set Flow Rate & Leak Check Record and a photocopy of the post sampling check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheets and forward the original anomaly report to the QA department.

10.3.2 Sample Screening

At the laboratory's discretion unknown samples may be screened prior to initial analysis to determine if the sample requires dilution. Unless otherwise requested by the client the laboratory does not provide screen data with the data package report even when primary dilutions are performed based on the results of the screen analysis.

To prepare a sample for screen analysis connect the sample canister to the autosampler connected to screening instrument and analyze 20 mL of sample. Acquire and evaluate the results. If the results of screen analysis indicate that a target compound is above its upper

range of calibration. Calculate a recommended dilution factor (DF) by dividing the concentration of analyte found by 30. Record the recommended DF on the screen worksheet.

NOTE: Samples are screened on a GC/MS instrument that is programmed with the operating conditions given in Section 10.4.1 of this SOP. The calibration is checked weekly with a single point calibration standard at a concentration of 10 ppbv for all target analytes. The calibration is checked more frequently when the results of instrument analysis do not correlate well with the results of the screen analysis.

10.3.3 Sample Dilutions & Pressure Adjustment

Field samples should be diluted prior to initial analysis when the screen results indicate that the concentrations are above calibration range and when the laboratory has sufficient knowledge of the sample (history) to know that the sample will require dilution. Field samples must be reanalyzed at a dilution initial analysis when the concentration of target compounds in initial analysis exceed of the upper range of calibration.

When the return negative pressure of a canister is greater than -15”Hg, make-up air is added to provide sufficient volume of make-up air in order to have an adequate sample volume for analysis. The addition of make-up air is considered a canister dilution.

To dilute the sample:

1) Attach the sample canister to the zero air line equipped with a pressure gauge that reads negative pressure in (“Hg) and positive pressure in (psig).

2) Ensure the valve of the zero air line is closed then open the valve of the sample canister. Record the negative pressure reading in the Canister Dilution Worksheet or on the canister’s tag.

3) Slowly open the valve of the zero air line and fill the canister until canister pressure gauge reads -10”Hg. Do not open the valve to such an extent that the zero air line pressure drops below 15 psig and do not allow the zero air line to reach equilibrium otherwise you will contaminate the zero air line.

4) When the desired pressure is achieved, close the canister valve and the valve on the zero air line; wait 15 seconds.

5) Open the canister valve and record the final pressure reading in psig.

6) Close the canister valve and remove the valve from the zero air line.

7) Record the initial and final pressure readings in the TALS canister dilution tracking module. If the final pressure is below ambient, the “HG reading must be converted to psig by dividing the value by 2 prior to entry into the TALS worksheet.

When the return pressure of a canister is positive, the pressure must be adjusted to near ambient (0”Hg) prior to analysis. To adjust the pressure to ambient, vent the canister to ambient in a fume hood by opening the canister value for ~4-5 seconds, close the valve. For higher

pressure canisters, open the valve and listen for a release of air then close the valve when the sound recedes.

If a trip blank is provided, pressurize the trip blank canister to 10 psig. The pressurization of the trip blank is not considered a dilution.

10.3.4 QC Sample Preparation

To prepare the method blank (MB): Fill a clean canister that has never been used to collect environmental samples and has never left the laboratory to 20 psig with zero air. Continue to use this canister as the MB until the pressure of the canister reaches 0 psig, at which time, recharge with zero air to 20 psig and reuse.

To prepare the LCS: Follow the instructions provided in Appendix B for preparation of the working ICV/LCS standard. If an LCSD and replicate precision is requested, the aliquot for the LCSD must be taken from the LCS canister. If an LCSD and duplicate precision is requested, prepare another LCS in a separate canister to serve as the LCSD.

10.4 Sample Analysis

10.4.1 Instrument Operating Conditions

Optimize the GC and MS conditions for compound separation and sensitivity.

The recommended operating conditions are as follows:

Thermal Desorb:	Initial Trap #1 Temperature: -110ºC Desorb Temperature from Trap #1 to #2: 0 ºC Total Volume Transfer by Mass Flow Controller: 40 mL Initial Trap #2 Temperature: -15 ºC Desorb Temperature from Trap #2 to #3: 200ºC Transfer time 3.5 minutes Initial Trap #3 Temperature: -165 ºC Injection Trap #3 Temperature: 70ºC Injection Time: 1.5 minutes Trap #3 Temperature after Injection: -165 ºC
Carrier Gas:	Helium, Ultra High Purity
Cryogenic Focusing Gas:	Liquid Nitrogen
Flow Rate:	~1.5 mL/min
Temperature Program:	Initial Temperature: 40ºC Initial Hold Time: 4 minutes Ramp1 Rate: 20ºC/min. to 200ºC. Ramp 2 Rate: 40ºC/min. to 220ºC Final Temperature: 220ºC Final Hold Time: 6.5 minutes
Electron Energy:	70 electron volts
Mass Range:	35-265 amu
Scan Time:	≥1 scan per second

These operating conditions may be changed but once the operating conditions are established for initial calibration the same conditions must be used until a new calibration is performed.

10.4.2 Analytical Sequence

An example analytical sequence that includes initial calibration (ICAL) is provided below. When ICAL is not performed, the sequence begins with the tune standard and is followed by the CCV, LCS, LCSD, and method blank. If sufficient time remains in the 24 hours analytical window after initial calibration, QC and field samples may be analyzed without the CCV and the ICV will serve as the LCS for the sequence. The MB, LCS and LSCD must be analyzed at a frequency of every 20 samples or with each analytical sequence whichever is more frequent.

1. Tune Standard (BFB)
2. ICAL
3. ICV
4. CCV
5. LCS (repeat every 20 samples)
6. LCSD (when requested)
7. MB (repeat every 20 samples)
8. Field Samples (including trip blanks)

Attach the canisters to the autosampler inlet in the order of the analytical sequence then initiate the analytical sequence. The autosampler introduces 200 mL of sample volume from each canister to the instrument system and adds 20 mL of the mixed gas standard to each sample.

Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system tentatively identifies target analytes by comparing the retention time of the peaks to the window set around the continuing calibration standard, and searches in that area for the primary ion and up to two secondary ions characteristic of the target analyte.

All tentative identifications made by the computer are reviewed and either accepted or rejected by the primary analyst. The identification made by the system is accepted when the following criteria are met:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP BR-QA-006 *Manual Integration*.

11.2 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the on-column concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

Final results are calculated in TALS.

11.3 Calculations

Analytical results are calculated as follows:

- **Dilution Factor**

$$DF = \frac{V_2}{V_1} x \frac{V_4}{V_3}$$

Where:
V_1 = Pre-Dilution Canister Volume
V_2 = Post-Dilution Canister Volume
V_3 = Sample Amount (mL)
V_4 = Base Sample Amount (200 mL)

- **Relative Response Factor (RRF)**

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Where:
A_x = Area of the quantitation ion of the analyte
A_{is} = Area of the quantitation ion of the internal standard

C_x = Concentration of analyte in concentration units (ppbv)
C_{is} = Concentration of internal standard in concentration units (ppbv)

- **Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{Mean} x100$$

Where:
SD = Standard deviation individual response factors
Mean = Average of five response factors

- **Sample Concentration**

$$C_x = \frac{(A_x)(C_{IS})}{(A_{IS})(\overline{RRF})}(DF)$$

Where:
C_x = Compound concentration (ppbv)
C_{IS} = Concentration of associated internal standard (ppbv)
A_{IS} = Area of quantitation ion for associated internal standard
A_x = Area of quantitation ion for compound
DF = Dilution Factor
Mean RRF = Average Relative Response Factor from initial calibration.

- **Unit Conversion from ppbv to ugm3**

$$\text{Analytical Result (ug/m3)} = \text{Result(ppbv)} \times \left(\frac{mw}{24.45}\right)$$

Where:
mw = molecular Weight

Example:
Benzene Result = 56 ppbv
Benzene mw = 78.108

$$\text{Analytical Result (ug/m3)} = 56\ \text{ppbv} \times \left(\frac{78.108}{24.45}\right)$$

Result(ug/m3) = 178.9 ug/m3 reported as 180 ug/m3

- **Percent Recovery (%R)**

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Where:
C_s = Concentration of the spiked sample (ppbv)
C_n = Nominal concentration of spike added (ppbv)

- **Precision (%RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

Where:
C_1 = Measured concentration of the first sample aliquot
C_2 = Measured concentration of the second sample aliquot

11.4 Data Review

11.4.1 Primary Review (Performed by Primary Analyst)

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the NCM is suspected, which action (if any) was taken and why and the outcome of the action taken.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Set the batch to 1st level review.

Record your review on the data review checklist.

11.4.2 Secondary Review (Performed by Peer Reviewer)

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements

were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP are were approved and are properly documented.

Spot-check 15% of samples in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Generate a “before” and “after” chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample.

- Generate the Manual Integration Summary Report. Document your review of manual integrations on the summary report and obtain any review signatures of integrations performed during secondary review as required.

  If the reviewer disagrees with the integration performed by the primary analyst, the secondary data reviewer should not change the integration. Instead, he/she should consult with the primary analyst that performed the integration and both the reviewer and the primary analyst should agree the integration should be changed. If consensus between the primary analyst and the peer reviewer cannot be achieved; both should consult with the Technical Manager or department management for resolution. Any changes to the integration should be performed by the primary analyst. If it is necessary for the secondary reviewer to perform the manual integration because the primary analyst is out of the office; the integration made by the peer reviewer must be reviewed by another peer reviewer or by department management to verify the integration was performed and documented in compliance to SOP BR-QA-005. If the original analyst that performed the integration is out of the office, the data reviewer may consult with the Department Manager (DM), Department Supervisor (DS) or the Technical Manager (TM) to verify the change he/she thinks is needed is warranted and should be made.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review.

Run the QC checker and fix any problems found. Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

Record second level review on the data review checklist.

11.5 Data Reporting

Data reporting and creation of the data deliverable is performed by TALS using the formatters set by the project manager during project initiation.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005,

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out:

- None

15.0 References / Cross-References

- EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-001 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- LaboratorySOP BR-QA-006 Procedures & Documentation Requirements for Manual Integration
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

Not Applicable.

17.0 Attachments

- Table 1: Target Compound List, RL, Internal Standard and Ion Assignments
- Table 2: Ion Abundance Criteria (BFB)
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables

18.0 Revision History

BR-AT-004r8, Revision 8:

- Title Page: Updated approval signatures
- All sections: Added procedure for ethanol
- Section 10: Changed calibration groups

Revision 7:
Section 11.4.1: Expanded on discussion of carry over.
Appendix C: Removed.
Appendix D: Removed.
Appendix E: Removed.

Table 1: Routine Compound List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dichlorodifluoromethane	75-71-8	0.5	5	85	87		1	B
Freon-22	75-45-6	0.5	5	51	67	69	1	B
1,2-Dichlorotetrafluoroethane	76-14-2	0.2	2	85	135	87	1	A
Chloromethane	74-87-3	0.5	5	50	52		1	B
n-Butane	106-97-8	0.5	5	43	41	58	1	B
Vinyl Chloride	75-01-4	0.04	0.40	62	64		1	D
1,3-Butadiene	106-99-0	0.5	5	54			1	B
Bromomethane	74-83-9	0.2	2	94	96		1	A
Chloroethane	75-00-3	0.5	5	64	66		1	B
Isopentane	78-78-4	0.2	2	43	57	56	1	A
Bromoethene (Vinyl Bromide)	593-60-2	0.2	2	106	108	81	1	A
Trichlorofluoromethane	75-69-4	0.2	2	101	103		1	A
Pentane	109-66-0	0.5	5	43	57	72	1	B
EthylEther	60-29-7	0.2	2	59	45	74	1	A
Acrolein	107-02-8	5	50	56	55	37	1	C
Freon TF	76-13-1	0.2	2	101	151	103	1	A
1,1-Dichloroethene	75-35-4	0.2	2	96	61	63	1	A
Acetone	67-64-1	5	50	43	58		1	C
Isopropyl Alcohol	67-63-0	5	50	45	43		1	C
Carbon Disulfide	75-15-0	0.5	5	76			1	B
3-Chloropropene (Allyl Chloride)	107-05-1	0.5	5	41	76		1	B
Acetonitrile	75-05-8	5	50	41	40	39	1	C
Methylene Chloride	75-09-2	0.5	5	49	84	86	1	B
tert-Butyl Alcohol	75-65-0	5	50	59	41	43	1	C
Methyl tert-Butyl Ether	1634-04-4	0.5	5	73	43		1	B
trans-1,2-Dichloroethene	156-60-5	0.2	2	61	96		1	A
n-Hexane	110-54-3	0.5	5	57	86		1	B
1,1-Dichloroethane	75-34-3	0.2	2	63	65	83	1	A
Methyl Ethyl Ketone	78-93-3	0.5	5	72	43		1	B
cis-1,2-Dichloroethene	156-59-2	0.2	2	96	98		1	A
Tetrahydrofuran	109-99-9	5	50	42	72		2	C
Chloroform	67-66-3	0.2	2	83	85		1	A
1,1,1-Trichloroethane	71-55-6	0.2	2	97	99	61	2	A
Cyclohexane	110-82-7	0.2	2	84	56		2	A
Carbon Tetrachloride	56-23-5	0.2	2	117	119		2	A
2,2,4-Trimethylpentane	540-84-1	0.2	2	57	41	43	2	A
1,2-Dichloroethene (total)	540-59-0	0.2	2	61	96		1	A
Benzene	71-43-2	0.2	2	78	77		2	A
1,2-Dichloroethane	107-06-2	0.2	2	62	98		2	A
n-Heptane	142-82-5	0.2	2	43	71		2	A
Trichloroethene	79-01-6	0.04	0.40	95	130	132	2	D
Methyl Methacrylate	80-62-6	0.5	5	69	41	39	2	B
1,2-Dichloropropane	78-87-5	0.2	2	63	41		2	A
1,4-Dioxane	123-91-1	5	50	88	58		2	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dibromomethane	74-95-3	0.2	2	174	93	172	2	A
Bromodichloromethane	75-27-4	0.2	2	83	85		2	A
cis-1,3-Dichloropropene	10061-01-5	0.2	2	75	110		2	A
Methyl Isobutyl Ketone	108-10-1	0.5	5	43	58		2	B
n-Octane	111-65-9	0.2	2	43	57	114	2	A
Toluene	108-88-3	0.2	2	92	91		3	A
trans-1,3-Dichloropropene	10061-02-6	0.2	2	75	110		2	A
1,1,2-Trichloroethane	79-00-5	0.2	2	83	97	85	3	A
Tetrachloroethene	127-18-4	0.04	0.40	166	168	129	3	D
Methyl Butyl Ketone	591-78-6	0.5	5	43	58		3	B
Dibromochloromethane	124-48-1	0.2	2	129	127		3	A
1,2-Dibromoethane	106-93-4	0.2	2	107	109		3	A
Nonane	111-84-2	0.2	2	57	71	128	3	A
Chlorobenzene	108-90-7	0.2	2	112	77	114	3	A
Ethylbenzene	100-41-4	0.2	2	91	106		3	A
Xylene (m,p)	1330-20-7	0.5	5	106	91		3	A
Xylene (o)	95-47-6	0.2	2	106	91		3	A
Styrene	100-42-5	0.2	2	104	78		3	A
Bromoform	75-25-2	0.2	2	173	175	171	3	A
Cumene	98-82-8	0.2	2	105	120	77	3	A
1,1,2,2-Tetrachloroethane	79-34-5	0.2	2	83	131	85	3	A
Xylene (total)	1330-20-7	0.2	2	106	91		3	A
n-Decane	124-18-5	0.5	5	57	71	142	3	B
n-Propylbenzene	103-65-1	0.2	2	91	120	92	3	A
1,2,3-Trichoropropane	96-18-4	0.5	5	75	110	112	3	B
4-Ethyltoluene	622-96-8	0.2	2	105	120		3	A
1,3,5-Trimethylbenzene	108-67-8	0.2	2	105	120		3	A
2-Chlorotoluene	95-49-8	0.2	2	91	63		3	A
tert-Butylbenzene	98-06-6	0.2	2	119	91	134	3	A
1,2,4-Trimethylbenzene	95-63-6	0.2	2	105	120		3	A
sec-Butylbenzene	135-98-8	0.2	2	105	134	91	3	A
4-Isopropyltoluene	99-87-6	0.2	2	119	134	91	3	A
1,3-Dichlorobenzene	541-73-1	0.2	2	146	111	148	3	A
1,4-Dichlorobenzene	106-46-7	0.2	2	146	111	148	3	A
n-Undecane	1120-21-4	5	50	57	71	156	3	C
Benzyl Chloride	100-44-7	0.2	2	91	126	65	3	A
n-Butylbenzene	104-51-8	0.2	2	91	134	92	3	A
1,2-Dichlorobenzene	95-50-1	0.2	2	146	111	148	3	A
n-Dodecane	112-40-3	5	50	57	71	170	3	C
1,2,4-Trichlorobenzene	120-82-1	0.5	5	180	182		3	B
1,3-Hexachlorobutadiene	87-68-3	0.2	2	225	223		3	A
Naphthalene	91-20-3	0.5	5	128			3	B
1,2,3-Trichlorobenzene	87-61-6	0.2	2	180	182	145	3	A
Propylene	115-07-1	5	50	41	42	39	1	C
Vinyl Acetate	108-05-4	5	50	43	86		1	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Ethyl Acetate	141-78-6	5	50	43	74		1	C
Ethanol	64-17-5	5	50	46	45		1	E
Bromochloromethane	74-97-5	NA	NA	128	49	130	1	NA
1,4-Difluorobenzene	540-36-3	NA	NA	114			2	NA
Chlorobenzene-d5	3114-55-4	NA	NA	117			3	NA

Table 2: Tune Standard Criteria

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of mass 95
75	30.0 to 66.0 percent of mass 95
95	Base Peak, 100 percent relative abundance
96	5.0 to 9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of mass 95
175	4.0 to 9.0 percent of mass 174
176	93.0 to 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

Table 3: TO15 QC Summary & Recommended Corrective Action

QC Check	Frequency	Acceptance Critera	Recommended Corrective Action
Tune Standard	Prior to calibration and every 24 hours	See Table 2	Correct Problem. Reanalyze. No sa without a valid tune.
ICAL	Prior to sample analysis and when CCV fails	RSD for each analyte ≤ 30% with 2 exceptions up to 40%	Correct problem and repeat calibrati
ICV	Once after each ICAL	%R for all analytes within 70-130	Correct Problem. Reanalyze, re-ma analyze. If that fails, re-make all sta calibration.
Retention Time Window	Once per ICAL	NA	NA
RRT	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct Problem. Repeat ICAL
CCV	Daily before sample analysis after tune standard	%D ≤ 30	Correct Problem. Reanalyze once. 10.2.5 for instruction.
LCS	Each batch or every 20 samples, whichever is sooner.	%R for all analytes within 70-130	Reanalyze LCS, re-prep and reanaly associated samples if sufficient sam If corrective action not successful, in report and qualify sample results.
LCSD	Per Client Request	RPD ≤ 25	Reanalyze LCSD, re-prep and reana associated samples if sufficient sam If corrective action is not successful, report and qualify sample results.
Method Blank	Each batch or every 20 samples, whichever is sooner.	No analytes detected above RL	Reanalyze along with associated sa same compounds found in blank are concentration found in the blank.
Internal Standard	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Inspect system for malfunction. Rea data.
Sample Duplicate	Per Client Request	RPD ≤ 25	Consult with PM. Reanalyze or qual

Appendix A: Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: Environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): An analytical standard gas mixture containing all target analytes and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8°C) or liquid argon (bp -185.7°C).

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

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.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): An analytical standard mixture containing all target analytes and internal standard compounds that are prepared from a source independent of the source of the initial calibration standards. The purpose of the ICV is to verify that the initial calibration is in control.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Internal Standards (IS): Non-target analytes that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). IS are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.

Laboratory Control Sample (LCS) – A QC sample of known composition spiked with analytes of interest. The LCS evaluates method performance and ability to successfully recover target analytes from a clean matrix. LCS recovery is typically expressed as percent recovery and provides a measure of accuracy. A LCSD is a duplicate LCS prepared and analyzed from a separate canister to provide a measure of replicate precision.

Method Blank (MB): A canister of humidified ultra pure zero air that is treated exactly as a sample. The MBLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL): 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 measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Gas Mixture: A Commercially purchased concentrated gas mixture containing one or more method analytes

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

Prepare all standards using the McMillan Company 80SD mass flow controller. Prepare the standard in zero air, demonstrated to be analyte free. Store the standard at ambient temperature. Unless otherwise specified, assign an expiration date of 30 days from date of preparation unless the parent standard expires earlier, in which case, use the earliest expiration date.

Intermediate Calibration Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Custom Calibration Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15 L Summa Canister Expiration Period 3months
This standard contains all the target analytes listed in table 1.

Working Calibration Standards

Parent Standard	Calibration Standard	Parent Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Cal Standard 20 ppbv	Cal Standard 0.2 ppbv	20	155	15.46	0.2
Cal Standard 20 ppbv	Cal Standard 0.5 ppbv	20	386	15.46	0.5
Intermediate Calibration Standard	Cal Standard 5 ppbv	200	386	15.46	5
Intermediate Calibration Standard	Cal Standard 10 ppbv	200	773	15.46	10
Intermediate Calibration Standard	Cal Standard 15 ppbv	200	1160	15.46	15
Intermediate Calibration Standard	Cal Standard 20 ppbv	200	1546	15.46	20
Intermediate Calibration Standard	Cal Standard 40 ppbv	200	3092	15.46	40

Prepare in 6 L Summa Canister Expiration Period 3 months
Each calibration standard contains all the analytes listed in table 1 at the above concentrations.

Initial Calibration Levels

Calibration Level	Working Calibration Standard	Volume Analyzed (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.2 ppbv	200	0.2
Calibration Level 2	Cal Standard 0.5 ppbv	200	0.5
Calibration Level 3	Cal Standard 5 ppbv	200	5
Calibration Level 4	Cal Standard 10 ppbv	200	10
Calibration Level 5	Cal Standard 15 ppbv	200	15
Calibration Level 6	Cal Standard 20 ppbv	200	20
Calibration Level 7	Cal Standard 40 ppbv	200	40
Calibration Level 8	Cal Standard 0.2 ppbv	40	0.04

Prepare in 6L Summa Canister

Intermediate ICV/LCS Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)

ICV Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Working ICV/LCS Standard

Parent Standard	Calibration Standard	Stock Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Intermediate ICV/LCS Standard	ICV Standard 10 ppbv	200	773	15.46	10

Prepare in 6L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Intermediate Ethanol Calibration Standard at 500ppbv/v

1) Fill a 44 ml VOA vial with VOA free water. Remove 197ul of water from the vial.
2) Add 197 ul of >99.5% Ethanol neat material
3) Cap and shake/roll vial for 1 minute
4) Inject 10ul of the prepared water/ethanol mix into a fully evacuated 15 liter summa canister
5) Pump the syringe plunger 5 times to insure complete transfer of material
6) Immediately fill the canister to 22 psig with zero air.

Calibration Level	Working Calibration Standard	Volume added (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.5 ppbv	124	5
Calibration Level 2	Cal Standard 5.0 ppbv	309	10
Calibration Level 3	Cal Standard 10ppbv	464	15
Calibration Level 4	Cal Standard 15 ppbv	618	20
Calibration Level 5	Cal Standard 20 ppbv	1237	40
Calibration Level 6	Cal Standard 40 ppbv	3092	100

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Screening for Volatile Organics by Headspace GC/FID

Approvals (Signature/Date):

William Rhoades 5/29/12
William Rhoades Date
Technical Manager

Adam W Alban 30 May 12
Adam Alban Date
Health & Safety Manager / Coordinator

5/30/12
John Morris Date
Quality Assurance Manager

Robert C Hanisch 5/30/12
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Volatiles screening analysis by Headspace GC/FID is done to establish the approximate concentration of the following analytes to determine a workable dilution for final analysis by GC/MS. This is a semi-quantitative technique.

1.2 Analytes - The analytes that are evaluated by this method are listed in Table 1. Other compounds can be detected, and if present, would enter into the screening outcome.

1.3 Detection Limits - Since this procedure is a screen to determine approximate concentrations, detection limits do not apply.

1.4 Approximate analytical time - 20 minutes per sample.

2.0 Summary of Method

An aliquot of sample is analyzed using a special headspace vial, or by mini-extraction using methanol as an extraction solvent. The extracts are then analyzed by the headspace-GC/FID. The data are then evaluated to determine an applicable dilution for GC/MS detection limits.

3.0 Definitions

Volatile Organics: Any purgeable organic compounds that chromatograph when the column is operated in the 25-260 degree C range.

4.0 Interferences

Carbon tetrachloride co-elutes with the fluorobenzene internal standard. When a sample is found to contain carbon tetrachloride, it must be screened on a GC/MS instrument.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must

review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Hewlett Packard 5890A Gas Chromatograph or equivalent

6.1.2 Supelco 20 mm LB2 Septa or equivalent

6.1.3 Hewlett Packard frosted quartz 2 mm ID injection port liner or equivalent

6.1.4 J & W Scientific fused silica megabore 30 m x 0.54 mm open tubular column with DB-624 liquid phase or equivalent

6.1.5 Hewlett Packard Flame Ionization Detector or equivalent

6.1.6 Instrument maintenance is described in Attachment A.

6.1.7 Tekmar 7000 NT

6.1.8 HP Chemstation Chromatographic Data System or equivalent

6.1.9 Compaq Personal Computer or equivalent

6.1.10 Centrifuge

6.2 Supplies

6.2.1 Sun 20 mL VOA vials with TFE-lined 24-40 solid caps or equivalent

6.2.2 Hewlett Packard 10 mL headspace vials part # 9301-0717 or equivalent

6.2.3 Hewlett Packard aluminum crimp caps 20 mm par t# 9301-0718 or equivalent

6.2.4 Hewlett Packard septa - 20 mm TFE-faced silicone part # 9301-0976 or equivalent

6.2.5 Hewlett Packard crimper for 20 mm caps part # 9301-0720 or equivalent

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent water – Analyte Free (D.I. water that has been boiled and purged with nitrogen.)

7.2 Reagent grade Sodium Chloride

7.3 High purity Methanol (purge and trap grade)

7.4 8240B primary VOC calibration standard. Refer to the MS VOA standards prep database for contents and concentrations.

7.5 Screening Internal Standard (IS): BFB and fluorobenzene in methanol at 200 ppm, prepared at TestAmerica Denver.

7.6 MSVOA-SCS prepared at TestAmerica Denver (Table 2).

7.7 MSVOA-LCS prepared at TestAmerica Denver (Table 3).

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The GC/MS volatile organic analysis SOP DV-MS-0010 has complete details. In brief, water samples for volatile organic analysis come in 40 mL septum-cap vials with no headspace. Soil samples normally include at least one portion of sample in 4 oz wide-mouth jars, which should be used rather than the portion in EnCores or sealed vials.

8.2 All samples to be screened by Headspace-GC/FID for final analysis by MS must be extracted and analyzed within 48 hours of sample receipt.

9.0 Quality Control

9.1 Quality control batches consist of 20 samples. For every 20 samples, one LCS and one Method Blank are prepared. Once a batch has 20 samples a new batch will need to be started.

9.2 Low Level Extraction Method Blank:

9.2.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

9.2.2 Add 5 mL of DI water to a salted headspace vial.

9.2.3 Using a 25 uL Hamilton syringe, add 5 uL of each of the screening IS standard.

9.2.4 Cap tightly.

9.3 Medium Level Extraction Method Blank:

9.3.1 Whenever matrices are extracted with methanol, whether they are medium level soils or wastes, a methanol method blank must be prepared.

9.3.2 Add 5.0 mL purge and trap grade methanol and 5 g of Ottawa sand to a 20 mL screw cap vial.

9.3.3 Add 4.0 uL MSVOA-SCS at 2,500 ug/mL.

9.3.4 Shake briefly.

9.3.5 Save the method blank extract in two 2.0 mL vials that are labeled “BLANK”, with the lot number and the date prepped.

9.3.6 The number of method blanks can be minimized by preparing all methanol extracts at the same time on a given day.

9.4 Low Level Extraction LCS:

9.4.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

9.4.2 Add 5 mL of DI water to a salted headspace vial.

9.4.3 Using a 25 uL Hamilton syringe, add 5 uL of the screening IS standard.

9.4.4 Add 5 uL of the MSVOA-LCS standard.

9.4.5 Cap tightly.

9.5 Medium Level LCS:

9.5.1 Weigh 5.0 g pre-baked Ottawa Sand in to a 20 mL screw cap vial.

9.5.2 Add 5.0 mL purge and trap methanol, 4.0 µL MSVOA-LCS at 2,500 ug/mL, and 40. µL MSVOA-SCS at 250 ug/mL. Note that different QC codes may have different spiking requirements. Be sure to check the instructions for each lot.

9.5.3 Shake for two minutes and centrifuge as necessary.

9.5.4 Save the LCS extract in two 2.0 mL screw top vials that are labeled to indicate the lot number and that they are LCS samples.

9.5.5 Some clients may require Duplicate LCS. In those circumstances, prepare a LCS duplicate with the LCS.

9.6 The results of the method blank and LCS are strictly for internal reference to ensure that the screening results are reliable enough for the lab’s own screening purposes. Strict SW-846 type quality control acceptance criteria do not apply.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Low Level Extraction Samples:

10.1.1.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

10.1.1.2 Add 5 mL of sample to a salted headspace vial.

10.1.1.3 Using a 25 uL Hamilton syringe, add 5 uL of the screening IS standard.

10.1.1.4 Cap tightly.

10.1.2 Low-level Extraction of Industrial Soils:

Note: If sample is not homogenous, mix with spatula to ensure homogeneity before use.

10.1.2.1 Weigh 2.0 to 2.5 g soil sample designated for screen analysis into a prebaked salted headspace vial.

10.1.2.2 Add 5 mL reagent water.

10.1.2.3 Add 5 uL screening IS (at 200 ug/mL). Cap tightly.

10.1.3 Medium-level Extraction of Solids and Wastes:

Note: If sample is not homogenous, mix with spatula to insure homogeneity before use.

10.1.3.1 Weigh 4.95 - 5.05 g soil designated for screen analysis into a 20 mL vial. Record in log book.

10.1.3.2 Add 4.95 mL methanol (add 4.90 mL methanol depending on method code). Recap tightly.

10.1.3.3 Add 4.0 uL MSVOA-SCS at 2,500 ug/mL.

10.1.3.4 Shake vigorously by hand for ~2 minutes.

10.1.3.5 Centrifuge at setting #4 for 3 to 5 minutes to separate solid matter.

10.1.3.6 If the sample absorbs a significant amount of methanol and there is less than 3 mL of free methanol:

- repeat the extraction with increasing amounts (5 mL increments) of methanol until there is enough methanol to fill at least one vial with no headspace plus a second vial one-half full
- add additional MSVOA-SCS standard in the proportion of 4 µL per 5 mL of methanol
- record the amount of methanol and standard in the logbook

10.1.3.7 Carefully draw the extract off the top, trying not to disturb the lower solid layer. Transfer the extract to two 2 mL screw top vials that have been labeled and taped (for GC/MS analysis). Fill vial A to the top, leaving as little headspace as possible. Fill vial B at least ½ full.

10.1.3.8 One vial is used for the screening analysis. The second is used for the determinative analysis by GC/MS.

10.1.4 Extraction of oils:

10.1.4.1 If miscible: Use 1 mL volumetrically, record the exact weight, and then add 4 mL of methanol.

10.1.4.2 If not miscible: Use approximately 1 g of sample and add 5 mL of methanol

10.2 Calibration

None

10.3 Sample Analysis

10.3.1 GC conditions temperatures and temperature programs are listed in Table 4.

10.3.2 19395A Headspace Sampler, Tekmar 700HT conditions are listed in Table 5.

10.3.3 Multichrom analysis

10.3.3.1 Determine sample order for each column and record it on the instrument log.

10.3.3.2 It is important that the analyst refer to the Multichrom Reference Manual Section 5, “Run Sequence File," pp. 77-113.

10.3.3.3 All runs will begin with a blank followed by the primary standard.

10.4 Start the run

10.4.1 Make sure the autosampler is in “Auto” mode. Set the autosampler to run the desired vial range by hitting the “A/S” button and changing the start and stop values as needed.

10.4.2 Make sure the instrument purges for A & B are off.

10.4.3 Make sure remote switches on Headspace Sampler on.

10.4.4 In the HP ChemStation software, from the “Sequence” pull-down menu, load the sequence file for the correct day and change the filename for the data file to the current day.

10.4.5 Under the sequence table option, enter the sample IDs in the sequence. For instrument I, choose the “8260SCRE” method. Choose “TEKMAR2” for instrument T.

10.4.6 After the sample IDs have been entered into the sequence and the vials are loaded on the autosampler, hit the "Start" button and then press "OK" to start the run.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

11.1.1 All identification is based on retention time. The internal standards (ISs) will reflect any fluctuations in retention time between runs.

11.1.2 Retention time windows for the first eight compounds (1,1-dichloroethene through carbon tetrachloride) are 3%. Windows for remaining compounds are 2.5%. Retention times must be reviewed daily with each daily calibration, updating the method if necessary. If changes are made, be sure to process samples with the updated method.

11.1.3 Review the daily calibration standard to insure that all peaks have been identified correctly. Except after column change or maintenance, retention times and areas should be similar to the previous daily calibration.

11.2 Estimating Dilution Levels Based on Screening Results

VOA screening analysis establishes the approximate concentration of target analytes to determine a dilution, which will place the highest concentration target compound at a level of 30 µg/mL or non-target compounds at a level of 60 µg/mL.

The objective of this process is to use a dilution factor that will put the GC/MS analytical results in the middle of the upper range of the calibration. Since the FID response is variable for the target compounds, the screening system is more effective for some classes of compounds than others.

The GC/MS analyst should evaluate one sample in a project at the recommended dilution determined from the following steps. This can be useful to evaluate screening information for other samples in the project.

11.2.1 Estimating Dilution Levels for Different Classes of Compounds

11.2.1.1 Since the FID response is variable for the target compounds, the screening system is more effective for some classes of compounds than others.

11.2.1.2 System is effective for Benzene, Toluene, Xylenes, and most hydrocarbons (good responders).

11.2.1.3 System is moderately effective for chlorinated compounds (moderate responders).

11.2.1.4 System is poor for ketones and trihalomethanes (poor responders).

11.2.1.5 System is not effective for acetone (does not extract well).

11.2.1.6 Dilutions based on moderate and poor responders can lead to over-dilution if the peak identifications are not correct. Because the identifications for bromoform, dibromochloromethane, and bromodichloromethane are not reliable with this screening method, dilutions should not be based on these compounds. See Table 6 for reduction of data.

11.2.2 Estimating Dilution Level Needed for Low-Level Waters

11.2.2.1 Determine the dilution factor by dividing the concentration of the highest analyte until it is approximately 30-45 ug/mL. For example, if a compound is present at 300 ug/mL, a 10x dilution is needed, and this will be obtained by diluting 2.0 mL of sample to 20 mL for the final analysis.

11.2.2.2 Unknowns are calculated using the response factor of 1,1-DCE for unknowns that elute before benzene. Meta- and para-xylenes are used for everything else. If any unknown is greater than the highest concentration target, the dilution recommendation is based on the unknown using the same calculation as above. If the identification of chlorinated compounds like chloroform or 2-chloroethyl vinyl ether is suspect, recalculate the compound as an unknown and present both results.

11.2.2.3 If no targets or unknowns are >20 ug/L in the sample, the recommended volume is 20 mL or 100%.

11.2.2.4 If the screen recommendation is <0.1 mL, serial dilute the sample and rescreen. See Table 6.

11.2.3 Estimating Dilution Levels for Low-level Soils

11.2.3.1 Use the same techniques and calculations as water samples in the previous section, substituting g for mL.

11.2.3.2 If screen indicates less than 0.5 g needed for industrial, or <1.0 g for AFCEE, prepare a medium level extract. See Table 6.

11.2.4 Estimating Dilution Levels for Medium-level Solids and Wastes

11.2.4.1 These extractions use 5 grams of soil in 5 mL of methanol. The optimum sample size for GC/MS = 0.100 mL methanol extract in 5 mL water (0.100 mL 5000 ug/L = 5.0 mL 100 ug/L)

11.2.4.2 Techniques and calculations are similar to water samples, except divide the highest concentration of a target compound by 2. The result is equal to the recommended volume of extract to add to 5 mL of water for GC/MS analysis.

11.2.4.3 Unknowns are calculated using the response factor of 1,1-DCE for unknowns that elute before benzene. Meta- and para-xylenes are used for everything else. If any unknown is greater than the highest concentration target, the dilution recommendation is based on the unknown using the same calculation as above. If

the identification of chlorinated compounds like chloroform or 2-chloroethyl vinyl ether is suspect, recalculate the compound as an unknown and present both results.

11.2.4.4 If the screen recommendation is <5 uL, serial dilute the methanol extract and rescreen. See Table 6.

11.3 Reporting Requirements

11.3.1 Screening results are not entered into the LIMS system.

11.3.2 The record in LIMS must be completed and released on the same day that samples are extracted. This is important for medium level methanol extractions because the test completion date is equal to the preparation test for GC/MS VOA tests. If a sample is re-extracted at a later date, the screen test completed test must be changed to reflect the latest extraction date.

11.3.3 All data should be analyzed, reduced, and reviewed by the day after extraction. Data from each project analyzed is grouped into separate folders.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no MDL study for a screening process.

12.2 Demonstration of Capabilities

Manual Initial Demonstration of Proficiency (IDOC) forms are used to document analyst proficiency initially and on-going on an annual basis.

12.3 Training Requirements

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.

13.0 Pollution Control

The volumes of methanol used for the medium-level extraction are kept as small as practical to minimize the generation of flammable and poisonous waste.

14.0 Waste Management

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 Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

14.1 Waste Streams Produced By This Procedure

The following waste streams are produced when this method is carried out.

- Methanol extract vial waste – Expired Extract Vials (A)

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and method revisions through Final Update IV, 2007. Determinative Chromatographic Separations, Methods 8000B, 8000C, and Nonhalogenated Organics by Gas Chromatography, Methods 8015B, 8015C and 8015D.

15.2 Manuals for Hewlett Packard Headspace Autosampler 19395A and Tekmar Dohrmann Headspace Autosampler 7000HT.

16.0 Method Modifications:

There are none for this procedure.

17.0 Attachments

Table 1: Analytes Determined by This Method
Table 2: MSVOA-SCS
Table 3: MSVOA-LCS
Table 4.1: GC Operating Conditions for Method 8260 SCR.M
Table 4.2: GC Operating Conditions for Method TEKMAR2.M
Table 5: GC and Sampler Conditions
Table 6: Dilution Determination from Screen Results
Attachment 1: Instrument Maintenance

18.0 Revision History

- Revision 3.4, dated May 31, 2012
  - Annual Technical Review
  - Grammatical, spelling and formatting changes throughout
- Revision 3.3, dated May 31, 2011
  - Revised the target weight for low-level extraction of industrial soils
  - Corrected table references
  - Expanded references to include method updates to SW-846
  - Grammatical, spelling and formatting changes throughout
- Revision 3.2, dated May 31, 2010
  - Annual review, only clerical changes made.
- Revision 3, dated 29 February 2008

- Integration for TestAmerica and STL operations.
- Method was updated to match current practices (throughout the SOP).
- Tables and attachments updated to current practice.

- Revision 2, dated 06 October 2004
  - STL Corporate Safety and Waste Management requirements are added to Sections 5 and 15, respectively.
  - Instrument maintenance is described in Attachment A.
  - The volumes and concentrations of spike mixes are changed.
  - QC sample instructions are moved to the QC section.
  - The operating conditions and instructions are given for current instrumentation, which are different than in the previous revision.
  - The target concentration for dilutions is changed to a lower concentration to match the current working range for our 8260B procedures.
  - Instructions about IDOCs documentation for screening analysts are added to Section 13.
- Revision 1, dated 11 February 2002
  - Changes from previous revision include reformatting for STL requirements, and inclusion of instrument conditions for a second screening instrument.

Table 1.

Analytes Determined by This Method

1,1,1-Trichloroethane	Chlorodibromomethane
1,1,2,2-Tetrachloroethane	Chloroform
1,1,2-Trichloro-1,1,2-trifluoroethane	Cis-1,2-dichloroethene
1,1,2-Trichloroethane	Cis-1,3-dichloropropene
1,1-Dichloroethane	Dibromoethane
1,1-Dichloroethene	Ethanol
1,2,3-Trichloropropane	Ethyl methacrylate
1,2-Dibromoethane	Ethylbenzene
1,2-Dichlorobenzene	Hexane
1,2-Dichloroethane	Iodomethane
1,2-Dichloropropane	m-Xylene
1,3-Dichlorobenzene	Methyl tert-butyl ether
1,4-Dichlorobenzene	Methylene chloride
1,4-Dioxane	Naphthalene
2-CLEVE	o-Xylene
2-Butanone	p-Xylene
2-Hexanone	Styrene
4-Methyl-2-pentanone	t-Butanol
Acetone	Tetrachloroethene
Benzene	Tetrahydrofuran
Bromodichloromethane	Toluene
Bromoform	Trans-1,2-dichloroethene
Carbon Disulfide	Trans-1,3-dichloropropene
Carbon tetrachloride	Trans-1,4-dichloro-2-butene
Chlorobenzene	Trichloroethene

Table 2.

MSVOA-SCS

250 ug/mL of following components in methanol

1,2-Dichloroethane-d4
4-Bromofluorobenzene (BFB)
Toluene-D8
Dibromofluoromethane

Table 3.

MSVOA-LCS

200 ug/mL of following components in methanol

Toluene
Trans-1,2-Dichloroethene
Trans-1,3-Dichloropropene
Trichloroethene
1,1-Dichloroethane
1,1-Dichloroethene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2-Chloroethyl Vinyl Ether
Benzene
Bromodichloromethane
Bromoform
Carbon Tetrachloride
Chlorobenzene
Chloroform
Cis-1,3-Dichloropropene
Dibromochloromethane
Ethylbenzene
Methylene Chloride
Tetrachloroethane

Table 4.1.

GC Operating Conditions for Method 8260 SCR.M

TABLE 4.1
Gas Chromatograph Operating Conditions
8260SCRE.M

Run Time Checklist			
Pre-Run Cmd/Macro	Off		
Data Acquisition	On		
Standard Data Analysis	Off		
Customized Data Analysis	Off		
Save GLP Data	Off		
Post Run Cmd/Macro	On		
Name	macro c:\gc_Itran.mac, GO		
Save Method with Data	Off		
Injection Source and Location			
Injection Source	Manual		
Injection Location	Front		
Oven/Det			
Runtime (min):	15.9		
Zone Temperatures			
	State	Setpoint (°C)	
Inlet A	Off	220	
Inlet B	Off	220	
Detector A	On	300	
Detector B	On	300	
Aux	Off	50	
Oven Zone			
Oven max	400 °C		
Equib Time	0.1 min		
Oven State	On		
Cryo State	Off		
Ambient	25 °C		
Cryo Blast	Off		
Oven Program			
	Setpoint		
Initial Temp	50 °C		
Initial Time	0.00 min		
Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	8.00	120	0.00
2 (A)	20.0	260	0.20
Purge Valve Settings			
Purge A/B			
	Init Value	On Time (min)	Off Time (min)
A (Valve 3)	Off	0.00	0.00
B (Valve 4)	Off	0.00	0.00
A - Splitless Injection	Yes		
B - Splitless Injection	Yes		
Valves/Relays Information			
Initial Setpoints			
5890 Valves			
	Valve 1	Off	
	Valve 2	Off	
	Valve 3 (Purge A)	Off	
	Valve 4 (Purge B)	Off	
Detector Information			
Detector A:			
Type	FID		
State	On		
Detector B:			
Type	FID		
State	On		
Signal Information			
Save Data	Signal 1		
Signal 1			
Signal	Det. A		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	35.00 min		
Signal 2			
Signal	Det B		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	35.00 min		

Table 4.2.

GC Operating Conditions for Method TEKMAR2.M

TABLE 4.2
Gas Chromatograph Operating Conditions
TEKMAR2.M

Run Time Checklist			
Pre-Run Cmd/Macro	Off		
Data Acquisition	On		
Standard Data Analysis	Off		
Customized Data Analysis	Off		
Save GLP Data	Off		
Post Run Cmd/Macro	On		
Name	macro c:\gc_Ttran.mac, GO		
Save Method with Data	Off		
Injection Source and Location			
Injection Source	Manual		
Injection Location	Front		
Oven/Det			
Runtime (min):	15.9		
Zone Temperatures			
	State	Setpoint (°C)	
Inlet A	On	220	
Inlet B	Off	50	
Detector A	On	330	
Detector B	Off	50	
Aux	Off	50	
Oven Zone			
Oven max	400 °C		
Equib Time	0.1 min		
Oven State	On		
Cryo State	Off		
Ambient	25 °C		
Cryo Blast	Off		
Oven Program			
	Setpoint		
Initial Temp	50 °C		
Initial Time	0.00 min		
Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	8.00	120	0.00
2 (A)	20.0	260	0.20
Purge Valve Settings			
Purge A/B			
	Init Value	On Time (min)	Off Time (min)
A (Valve 3)	Off	0.00	0.01
B (Valve 4)	Off	0.00	0.01
A - Splitless Injection	No		
B - Splitless Injection	No		
Valves/Relays Information			
Initial Setpoints			
5890 Valves			
	Valve 1	Off	
	Valve 2	Off	
	Valve 3 (Purge A)	On	
	Valve 4 (Purge B)	On	
Detector Information			
Detector A:			
Type	FID		
State	On		
Signal Information			
Save Data	Signal 1		
Signal 1			
Signal	Det. A		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	650.00 min		
Signal 2			
Signal	Testplot		
Data Rate	5.000 Hz		
Peakwidth	0.053min		
Start Time	0.00 min		
Stop Time	650.00 min		

Table 5.
GC and Sampler Conditions

GC	Conditions
Sample Loop	1 mL Loop
Platen Temperature	85°C
Platen Equilibration Time	0 minute
Sample Equilibration Time	30 minutes
Vial size	22 mL
Mixer	On
Mixing Time	2 minutes
Mix Power	5
Stabilize Time	2 minutes
Cryo Cool down / Minutes at	NI
Pressurize Setting	6 psi at 40 mL/min
Pressurize Time	0.3 minute
Pressurize Equilibration Time	0.05 minute
Loop Fill Time	0.3 minute
Loop Equilibrium Time	0.05 minute
Inject	1 minute
Cryo Inject / Minutes at	NI
Valve Temperature	85°C
Line Temperature	85°C
Cryo Union Heater	NI
Injections Per Vial	1
GC Cycle Time	42 minutes
Parameter Optimization	Off
Detector	FID
Column	DB-624, 60M, 0.53 um
Carrier Flow	6 cc/min
Initial Temperature	35°C for 5 minutes
Rate	3°C/min to 100°C
Hold Time	1 minute

Tekmar 7 - HT	Sampler Conditions
Platen plate	85°C
Sample Loop	200°C
Sample Line	200°C
Sample Equilibration	7 minutes
Pressurize	0.5 minute
Pressurize Equilibration	0.1 minute
Loopfill time	0.5 minute
Loop Equilibration	0.2 minute
Inject	0.5 minute

Table 6.

Dilution Determination from Screening Results

The following table lists minimum and maximum sample size for various VOA tests based on results of the screening analysis.

TYPE	MATRIX	PREP	GC/MS MIN RUN	GC/MS MAX RUN	COMMENTS
Industrial	Water	LL-1.0 mL/5.0mL	DILN	20 mL	If <0.1 mL, serial dilute
Industrial	LL Soil	LL-1.0 g/5.0 mL	1.0 g	5.0 g	If <1 g, go to ML prep
Industrial	ML soil	ML-5.0 g/5.0 mL	DILN	100 uL	Standard Prod. If <5uL, serial dilute
Industrial	Waste	ML-5.0 g/5.0 mL	DILN	100 uL	If <5 uL, serial dilute
Industrial	TCLP	LL-100 uL/5.0 mL	DILN	1.0 mL	May do oil and water phase
AFCEE	Soil	ML-5.0 g/5.0 mL	1.0 g	5.0 g	If <1.0 g, go to ML prep
AFCEE	Soil	ML-5.0 g/5.0 mL	DILN	500 uL	If <5 uL, serial dilute

LL = Low level
ML = Medium level

Attachment 1.

Instrument Maintenance (Part 1)

1.0 Semi-annual Instrument Maintenance

1.1 Change septa. See HP instrument manual for instructions

1.2 Check flows. Optimum flows are:

Helium 12 mL/min
Nitrogen 10 mL/min
Air 370 mL/min
Hydrogen 30 mL/min
Adjust if necessary.

1.3 Cut the column

1.3.1 Remove fitting on injection port end of column.

1.3.2 Unwrap column one revolution and using a glass scorer, cut ~2 inches of column from injection port end. Inspect the cut end to be sure the cut was clean and square, leaving no jagged ends.

1.3.3 Replace the connecting nut and place a new 0.8 mm graphite/vespel ferrule on the end flush with the nut.

1.3.4 Using a ruler, measure 1 cm from the cut end of the column to the top of the nut thread. Mark the column under the nut so that when it is put in place in the injection port liner, the column amount measured extends up into the injection port liner. Turn the connecting fitting nut finger tight, then turn 3/4 to a full turn with a 3/16" wrench.

1.3.5 When cutting the detector end of the column, follow steps 1.3.1 through 1.3.3, applying to detector end.

1.3.6 When reconnecting detector end, feed column into detector as far as it will go, connect with nut, and withdraw 0.5 cm column out. Finish connection as in 8.2.4.4.

Attachment 1.

Instrument Maintenance (Part 2)

1.4 Annual Instrument Maintenance

1.4.1 Lower injection port temperatures and detector temperatures to ambient

1.4.2 Change injection port liners

1.4.2.1 Remove H.S. transfer lines. Be careful not to bend needle end. **CAUTION** - transfer line is hot.

1.4.2.2 Remove lower injection port nut

1.4.2.3 Carefully remove injection port liner and replace with freshly silanized liner.

1.4.2.4 Reassemble

1.4.3 Silanizing injection port liners

1.4.3.1 Place dirty injection port liners in acid soak for 1 hour minimum. Remove.

1.4.3.2 Rinse first with cold water, then hot water, then liberally with acetone, hexane, methylene chloride, and methanol. Let dry thoroughly.

1.4.3.3 Soak injection port liners in a 10% solution of Hexamethyldisilazane in Hexane for a minimum of 8 hours. Remove, rinse with 200-300 mL Hexane, and let dry.

1.4.4 Visually inspect FIDs for contamination by removing housing. If dirty, remove parts and sonicate in applicable solvent. Reassemble.

1.4.5 Restore operating temperatures.

Helpful hints: If normal signals of 2 to 5 cannot be achieved after maintenance, bake oven at 260^{O}C for 1-2 hours, then run 2 to 3 solvent blanks. This usually restores the signal.

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Title: Determination of Volatile Organics by GC/MS [8260B and 624]

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1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1. Additional analytes that can be determined by this SOP are listed in Tables 2, 3 and 4.

1.2 This SOP is applicable to Method 8260B, which is appropriate for compliance testing under RCRA regulations and Method 624 (CWA compliance testing). It is important that the procedural differences described in this document for these methods are carefully observed.

1.3 Appendix A of this SOP contains the modifications needed to run the instrument in the selected ion monitoring mode.

1.4 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.

1.5 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 60 μg/L for 8260B waters, 2.5 to 200 μg/kg for low-level soils, and 200 to 30,000 μg/kg for medium-level soils. The working range for Method 624 (5 mL purge) is 5-200 μg/L.

1.6 Reporting limits for Method 8260B are listed in Tables 1, 2, and 3. Reporting limits for Method 624 and 8260B SIM are given in Table A1 and Table Ap-1, respectively. Reporting limits for soil samples prepared by the AK methanol technique are listed in Table Bp-1.

1.7 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

2.0 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 gas 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 preserved in water (with or without sodium bisulfate) 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 is 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 sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed 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.

3.0 Definitions

3.1 Terms

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and in SOP DV-QA-003P, Quality Assurance Program.

3.2 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and percent drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.3 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.4 Initial Calibration Verification (ICV)

The ICV is a second-source calibration verification standard. In this SOP, the LCS and the MS/MSD spikes are second-source standards.

3.5 Continuing Calibration Verification (CCV)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.6 Selected Ion Monitoring (SIM)

Operation of the mass spectrometer in the selected ion monitoring mode to optimize the quantitative information at the expense of qualitative information gained from other methods of analysis.

4.0 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 co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the 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 (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample. (See Section 10.7.4.12.)

4.6 Interferences are observed with the surrogate Toluene-d_8 when the samples appear to be treated with potassium permanganate.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.1.2 Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 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.

6.1.3 Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax / Silica gel / Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.

6.1.4 Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.

6.1.5 Sample Heater: A heater capable of maintaining the purge device at 40 °C is necessary for low level soil analysis.

6.1.6 Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.1.7 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.1.8 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.1.8.1 Column 1: 60 m X 0.25 ID DB-624 with 1.4 µm film thickness.

6.1.8.2 Column 2: 75 m X 0.53 ID DB-624 wide bore with 3 µm film thickness.

6.1.9 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 of 4-bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.1.10 GC/MS interface: In general, glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.1.11 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. In addition, 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.

6.2 Computer Software and Hardware

6.2.1 Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Microsyringes: 10 µL and larger, 0.006-inch ID needle.

6.3.2 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.3.3 Balance: Analytical balance capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.3.4 Vials: 2 mL, 20 mL, and 40 mL with screw caps and Teflon liners

6.3.5 Disposable magnetic stirrers for low-level soil analyses

6.3.6 Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.3.7 Spatula: Stainless steel.

6.3.8 Disposable pipettes: Pasteur.

6.3.9 pH paper: Wide range.

6.3.10 Gases:

6.3.10.1 Helium: Ultra high purity, grade 5, 99.999%.

6.3.10.2 Compressed nitrogen: Used for instrument pneumatics.

7.0 Reagents and Standards

7.1 Methanol: Purge and Trap Grade, High Purity

7.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.3 Sand: Reagent grade Ottawa sand or equivalent.

7.4 Antifoam B, Silicon Emulsion, J. T. Baker, 100% purity.

7.5 Sodium bisulfate ($NaHSO_4$), reagent grade

7.6 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers' expiration date.

7.7 Calibration Stock Standard 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. Stock standards and aliquots for gases must be replaced at least every week. The Gas Standards Tracking Log is used to verify track open dates to assist in weekly replacement of the gas standards. See Attachment 1. Other stock standards must be replaced at least every 6 months.

7.8 Calibration Working standards

A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. 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.

7.9 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.10 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Tables 7 and 7A for internal standard components.

7.11 Surrogate Standards: Refer to Tables 8 and 8A for surrogate standard components and spiking levels.

7.12 Laboratory Control Sample Spiking Solutions: Refer to Table 10 for LCS components and spiking levels.

7.13 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 10.

7.14 Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of BFB in methanol is prepared from stock standards as described in Sections 7.7 and 7.8.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples

8.1.1 Water samples are collected in triplicate in 40 mL glass VOA vials with PTFE-lined septum caps with minimal headspace. There should be no bubbles present in the container larger than ~6 mm.

8.1.2 Preservation depends upon the target analytes and the sampling location. At a minimum, aqueous samples are stored refrigerated at ≤ 6 ^{o}C and not frozen. Specific preservation requirements are given in the following table. If multiple analytes are requested, it may be necessary to provide aliquots with different preservations. For each preservation technique, the samples should be collected in triplicate.

8.1.3 The State of Colorado Attorney General's office issued a letter on July 1, 1998 requiring that all samples collected for analysis of volatile organic compounds in groundwater must be collected without acid preservation. The letter explains that this is done to avoid effervescence with alkaline samples and loss of volatiles. The letter also explains that the holding time for unpreserved ground waters is 14 days.

8.1.4 SW-846 states that if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. The holding time for these unpreserved samples is 7 days. SW-846 does not otherwise provide guidance for processing unpreserved samples. EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

Preservation and Holding Time for Volatiles in Water

Analyte(s)	Reference	Preservation[1]	Holding time	Dechlorination Required[2]
Routine target analytes[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	SW-846, Ch. 4	Cool, ≤6ºC	7 days	Y
	624	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	624	Cool, ≤6ºC	7 days	Y
Acrolein[4]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	603	Cool, ≤6ºC (no HCl)	3 days	Y
	603	Cool, ≤6ºC, pH 4-5	14 days	Y
Acrylonitrile[4]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	603	Cool, ≤6ºC (no HCl)	14 days	Y
	603	Cool, ≤6ºC, pH 4-5	14 days	Y
2-Chloroethylvinyl ether (2-CLEVE)[5]	SW-846, Ch. 4	Cool, ≤6ºC (no HCl)	7 days	Y
	624	Cool, ≤6ºC (no HCl)	14 days	Y

[1] See Section 8.1.3 for samples collected in Colorado and Section 8.1.4 for samples to be analyzed by Method 8260B that are unpreserved.
[2] If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added
[3] Separate aliquots must be collected and preserved as indicated if acrolein, acrylonitrile, 2-CLEVE (by Methods 8260B or 624), vinyl chloride (by Method 8260B) or styrene (by Method 8260B) are also to be analyzed. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary.
[4] According to the source methods, the preferred method for acrolein and acrylonitrile is Method 603. In the Method Update Rule published in the Federal Register on May 18, 2012 (40 CFR Parts 136, 260, et. al.) EPA approved Method 624 for the determination of acrolein and acrylonitrile in wastewater. The current sample preservation and holding time requirements for acrolein and acrylonitrile apply to these compounds when analyzed by Method 624. Implementation of this rule is subject to individual state program decisions and timetables.
[5] SW-846 includes vinyl chloride and styrene in the list of compounds which require unpreserved sample for analysis. Method 624 does not include these two analytes on the standard analyte list.

8.2 Soil Samples

8.2.1 Soil samples can be taken using the EnCore™ sampler. Typically three Encores are collected per sampling location. At specific client request, unpreserved soil samples may be accepted for preservation at the lab.

8.2.1.1 Samples sent in the EnCore™ sampler to the lab for preservation must be preserved within 48 hours of sampling. They are preserved by extruding one sample into a clean VOA vial containing methanol for medium level analysis. The remaining two samples are extruded into vials containing water or sodium bisulfate ($NaHSO_4$) and water for low level analysis.

8.2.1.2 Samples are stored frozen after transfer from the EnCore™ sampler.

8.2.2 The more common way to collect soils is with Terra Core kits. Typically three aliquots are collected. Terra Core kits consist of the Terra Core sampling device and three 40 mL tared VOA vials. There are several ways to preserve the samples once sampled.

8.2.2.1 The samples collected with the Terra Core sampling device are extruded into empty vials and frozen in the field. The lab freezes the samples on receipt. These samples have a 14 day holding time from sampling.

8.2.2.2 The samples can be extruded into empty vials and shipped to the lab refrigerated. The lab freezes the samples within 48 hours of collection and the holding time is extended to 14 days from collection. The lab has the option to prepare the samples upon receipt by the addition of methanol to one vial and water or sodium bisulfate ($NaHSO_4$) and water to the remaining two vials. The samples are then refrigerated. The holding time for this latter preservation is 14 days from sampling.

8.2.2.3 Alternatively, the project team can request for each sample one tared vial containing methanol for medium level analysis and two tared vials containing water or sodium bisulfate and water, depending upon project requirements. An aliquot of the sample is extruded into each prepared vial while in the field and shipped on ice. The samples are refrigerated upon receipt at the laboratory. The holding time is 14 days from sampling for this field preservation technique.

8.2.3 Unpreserved Soils

8.2.3.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the laboratory. This is the old procedure based on Method 5030A. It is no longer included in subsequent revisions of Method 5030 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.2.3.2 The maximum holding time is 14 days from sampling until the sample is analyzed. Unpreserved samples should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative.

8.2.4 An additional bottle of unpreserved soil for each sampling location must be shipped for percent moisture determination.

8.2.5 A second bottle of unpreserved soil is sent for screening.

8.2.6 Preservation and holding times for volatiles in soils are summarized in the following table, based on SW-846 Method 5035A. The "Coring Tool" listed in the container column may be the EnCore™ or Terra Core sampler.

Preservation and Holding Time for Volatiles in Soil
Method 5035A

Container/Contents[1]	Preservation	Holding time	Analysis
Empty Sealed Vial	Freeze on-site to -7°C (do not freeze below -20°C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C	48 hours	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Frozen upon receipt at lab (< -7 °C, do not freeze below -20 °C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Preserved with methanol upon receipt at lab	14 days	Medium Level
Encore™ sampler used for transport	Cool to ≤ 6 °C or Freeze to < -7 °C in field	48 hours	Low or medium level
Encore™ sampler used for transport	Cool to ≤ 6°C or freeze to < -7°C in field and upon receipt at lab extruded to a sealed vial and either frozen to < -7°C or chemically preserved	14 days	Low or medium level
Vial containing reagent water	Sample is extruded into vial and frozen to < -7°C in field and maintained frozen upon receipt by the laboratory.	14 days	Low level
Vial containing reagent water	Sample is extruded into vial and cooled to ≤ 6°C in field then frozen to < -7°C upon laboratory receipt (within 48 hours of sampling).	14 days	Low level
Vial containing reagent water and 1 g $NaHSO_4$[2]	Sample is extruded into vial with preservative and cooled to ≤ 6°C. Stored at ≤ 6°C upon laboratory receipt.	14 days	Low Level
Vial containing methanol	Sample is extruded into vial with preservative, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

[1] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

[2] Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible

8.3 Trip blanks, consisting of laboratory prepared water samples with acid preservative, are also provided when bottles are supplied by the laboratory to the field. Trip blanks are used for both water and soil samples to monitor potential contamination from volatile compounds in transit and in the field.

8.4 A holding blank is stored in each refrigerator with the samples. This is analyzed every 7 – 14 days (see SOP DV-QA-0013).

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples (method blank, lab control sample, and matrix spike/matrix spike duplicate), processed using the same procedures and reagents within the same time period. 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. A method blank must be run on each instrument. See Policy DV-QA-003P for further details.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is Ottawa sand. For medium-level volatiles, the method blank consists of 5.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half 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.

The method blank must have acceptable surrogate recoveries. (See Section 9.4)

Corrective Actions: If the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone), 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.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than one-half 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.

If surrogate recoveries in the blank 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.

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 the narrative to provide further documentation.

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 Tables 8 and 8A.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean. Surrogate recovery limits are updated semi-annually and stored in the LIMS.

Corrective Actions: If any surrogates are outside 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 if either of the above checks reveal a problem.
- Re-prepare 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 necessary to re-prepare/reanalyze a sample only 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.

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 re-preparation/reanalysis 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.

9.5 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS spiking solution is prepared from a different source than are the calibration standards. The LCS contains a representative subset of the analytes of interest (See Table 10), and must contain the same analytes as the matrix spike. For low-level volatiles in water, the LCS matrix is reagent water. For low-level volatiles in soil, the LCS matrix is Ottawa sand.

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated semi-annually and stored in the LIMS

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAC, AFCEE, and the DOE. DoD requires individual project approval for the use of marginal exceedances. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Additional criteria are stated in the North Carolina QAS.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QSAS's in the public folders for the current requirements.

Corrective Actions: If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation 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. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

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 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 10. 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.

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated semi-annually and stored in the LIMS.

Corrective Actions: 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 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 reanalysis of the batch.

If an MS/MSD is not possible due to limited sample, then an LCS duplicate should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 Acid Preservation or pH adjustment

The stability of 2-chloroethylvinylether, acrolein, and according to the regulations, acrylonitrile is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed routinely from preserved VOA vials and since there is no reasonable way to achieve pH between 4 and 5, it is recommended that unpreserved vials be used for the analysis of these compounds.

Acceptance Criteria: To ensure detection of these compounds, samples must be processed correctly. Where Method 624 is being used for compliance purposes, the regulatory hold times take precedence.

Corrective Actions: If 624 data are not being generated for compliance purposes, the technical stability of the compounds may be considered. Where

method 8260 is the base method, it is allowable to qualify the results as estimated. To deviate from the regulatory hold times, the following documentation must be maintained:

- A NCM must be generated by the lab that the samples are for non-compliance.
- A NCM must be generated that results are not method compliant.

9.8 2012 MUR Required QC Elements

The May 2012 EPA Method Update Rule (MUR) to 40 CFR Part 136 for compliance testing under the Clean Water Act (CWA) requires laboratories to include 12 QC elements when performing the published or approved methods. See Work Instruction WI-DV-0060, QC Requirements for Methods Designated in 40 CFR Part 136, for list of approved test procedures performed by TestAmerica-Denver and the required QC elements in each of these methods.

10.0 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 an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preservation using EnCore™ Samplers.

10.3.1 Preservation in Methanol (Medium-Level Analysis)

10.3.1.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it on the label. Quickly add 5 mL of methanol and cap the vial.

10.3.1.2 If sufficient samplers are provided (or for the sample(s) designated by the client), prepare MS and MSD samples as above.

10.3.1.3 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.3.1.4 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.2 Preservation in Water (Low-Level Analysis)

10.3.2.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it on the label. Quickly add 5 mL of water and a magnetic stirrer. Cap the vial. Repeat for the remaining aliquot.

10.3.2.2 If requested by the client, 1 g of sodium bisulfate is added to the second sample preserved with water.

10.3.2.3 If sufficient samplers are provided for a sample in the batch, or for any samples identified by the client, prepare MS and MSD samples as above.

10.3.2.4 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of water to the blank. For the LCS, the volume of water added is dependent upon the spike list. Add 4.95 mL water if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Add a magnetic stirrer. Cap tightly. Store with the samples.

10.3.2.5 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.3 Screen the samples. (See Section 10.5.) If the screen indicates any samples will be analyzed as medium level, go to Section 10.5.1. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.4 Sample Storage for Field Preserved Samples

10.4.1 Obtain the weight of the soil added to each vial and record it in TALS.

10.4.1.1 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials for each analysis method (medium-level and low-level).

10.4.1.1.1 For the medium level method add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.4.1.1.2 For the low level method add water instead of methanol using the same volumes as in Section 10.4.1.1.1.

10.4.1.2 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.4.2 Screen the samples. (See Section 10.5) If the screen indicates any samples will be analyzed as medium level, go to Section 10.7.5. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.5 Sample Screening

10.5.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. See SOP DV-MS-0009. Alternatively, an appropriate aliquot can be determined from sample histories.

10.6 Sample Preparation for Medium-Level Analysis – Field or Lab Preserved

10.6.1 For each of the samples that are determined to be Medium-Level samples by the screening procedure, add the correct amount of surrogate spiking mixture for a final concentration of 2 μg/mL. Example: 4 μL of 2500 μg/mL for a nominal 5 g sample or 20 μg/mL for a nominal 25 g sample. Cap the sample vial. Surrogates are added to all QC samples as well as field samples.

10.6.2 Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples for a final concentration of 2 μg/mL.

10.6.3 Add the correct amount of matrix spiking solution to the LCS sample for a final concentration of 2 μg/mL. If 25 g samples are being used, adjust the proportions for the LCS accordingly.

10.6.4 Shake the samples for two minutes to distribute the methanol throughout the soil.

10.6.5 Centrifuge the samples to clarify the extract.

10.6.6 Remove a portion of methanol and store in a clean Teflon-capped vial with no headspace refrigerated at $\leq 6\ ^{o}C$ until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

10.7 Sample Analysis Procedure

10.7.1 All analysis conditions for samples must be the same as for the initial and continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

10.7.2 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 method blank, an LCS, and a MS/MSD.

10.7.2.1 If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. The

12-hour tuning requirements in Section 10.7.12.3 and 12-hour continuing calibration requirements in 10.7.14 must still be met. However, if any re-tuning or recalibration of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new QC batch must be started. For high-level soils the batch is defined at the sample preparation stage.

10.7.2.2 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.

10.7.2.3 Any reruns must be part of a valid batch. If dilutions of a sample are analyzed in the same calibration event they do not count towards the maximum batch count. (See DV-QA-003P.)

10.7.3 Water Samples

10.7.3.1 Purge-and-trap units that sample from a VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.3.2 All samples and standard solutions must be at ambient temperature before analysis.

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 be analyzed as soils.

10.7.3.3 To transfer a sample from its original container, fill a gas-tight syringe with the sample and adjust the sample volume based on the requested method. Place the measured sample into a clean VOA vial.

10.7.3.3.1 For Method 8260, 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.) Sample aliquots are measured in 25 mL gas tight syringes. Separate syringes are used for each sample.

10.7.3.3.2 For Method 624, 5 or 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.). Sample aliquots are measured in 5 or 25 mL gas-tight syringes. Separate syringes are used for each sample.

10.7.4 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.

10.7.4.1 For dilutions of aqueous samples which require less than 1 mL of sample the sample volume is added to 20 mL of reagent water in a VOA vial or in a gas-tight syringe.

10.7.4.2 For dilutions of aqueous samples which require more than 1 mL of sample, the volume of reagent water is adjusted so that the total volume of sample and reagent water is 20 mL. The dilution is made in the VOA vial by adding the appropriate amount of reagent water to the vial. The sample aliquot is then added to the closed vial by injecting below the surface of the water.

10.7.4.3 If the dilution required would use less than 5 µL of sample, then serial dilutions must be made in volumetric flasks.

10.7.4.4 Check and document the pH of the remaining sample. Document the pH value on the run log. If the pH is not as expected, based on the sample type and preservation, document in an NCM in the LIMS.

10.7.4.5 Sample remaining in the vial after sampling is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

10.7.4.6 For TCLP samples, use 2.0 mL of TCLP leachate and spike it with 2.5 µL of the 40 µg/mL TCLP spiking solution. Bring to a volume of 20 mL with reagent water.

10.7.4.7 Surrogates and internal standards are added to each sample at the instrument at the time of purging.

10.7.4.8 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe needle through the septum into the water. Surrogates and internal standards are added to these samples by the instrument.

10.7.4.9 Purge the sample for eleven minutes (the trap should be below 50 ºC).

10.7.4.10 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 2-5 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

10.7.4.11 Desorb time, bake time, and temperature are optimized for the type of trap in use. Some programs or clients have special requirements for the desorb time. Method 624 requires a 4 minute desorb time.

NOTE: The same conditions must be used for samples and standards.

10.7.4.12 If foaming of the sample occurs, reanalyze the sample with the addition of 1 µL of an antifoaming agent such as Antifoam B (J. T. Baker). A method blank spiked with 1 µL of the Antifoam B must also be analyzed with the sample. Document in an NCM.

10.7.5 Methanol Extracts of Soils

10.7.5.1 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 (typically 5 mL).

10.7.5.2 Add no more than 25 µL of methanolic extract (from Section 10.3.1 or 10.5.1) to the syringe for each sample and QC sample. Add surrogates to each sample.

10.7.5.3 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe through the septum of the vial.

10.7.5.4 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe.

10.7.5.5 Only internal standards are added at the instrument for methanol extracts.

10.7.5.6 Load the sample onto the purge and trap device and analyze as for aqueous samples. (See Section 10.7.3.)

10.7.6 Liquid Wastes that are Soluble in Methanol and Insoluble in Water

10.7.6.1 Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

10.7.6.2 Quickly add 7 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.

10.7.6.3 For an MS/MSD pair, add 6 mL of methanol to 2 mL of the sample in a tared vial. Add 1 mL of surrogate solution and 1 mL of matrix spike solution.

10.7.6.4 Prepare an LCS by adding 1 mL of surrogate solution and 1 mL of matrix spike solution to 8 mL of methanol.

10.7.6.5 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.

10.7.6.6 Add no more than 25 µL of methanolic extract (Section 10.7.6.2) to the syringe. Add internal standard (if used).

10.7.6.7 Load the sample onto the purge and trap device and analyze as for aqueous samples using 5 mL reagent water.

10.7.6.8 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe. (See Section 10.7.4.)

10.7.7 Low-Level Soil Sample Analysis following SW846 Method 5035

10.7.7.1 This technique is to be used when samples are collected utilizing SW-846 Method 5035. Pre-weighed vials are used to collect approximately a 5 gram aliquot of soil (see section 8.2).

10.7.7.2 Purge-and-trap units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.7.3 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.

10.7.7.4 The autosampler will heat and stir each sample as it is purged.

10.7.7.5 If any target analytes exceed the calibration range, analysis of the methanol preserved sample must be performed.

10.7.8 Low-Level Solids Analysis When Field Samples are Provided in a Jar

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.

10.7.8.1 This method is based on purging a heated sediment/soil sample mixed with water and, if applicable, internal and 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 ºC.

10.7.8.2 Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

10.7.8.3 Weigh out 5 g (or other appropriate aliquot) of sample into a clean VOA vial. Record the weight to the nearest 0.1 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 10.7.5 with preparation described in Section 10.5.1.

10.7.8.4 Rinse a 5 mL gas-tight syringe with organic-free water, and fill. Compress to 5 mL. Inject the spiked water into the VOA vial that contains the soil sample and add a stirring bar.

10.7.8.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

10.7.8.6 Prepare a Method Blank and LCS using 5 g of Ottawa sand and 5 mL of water. Add a stirring bar to each. Prepare the MS/MSD (based on

the sample requested by the client. The LCS spiking solution is added via a syringe inserted through the septum of the vial to the LCS and MS/MSD samples.

10.7.8.7 Low level soil samples may be analyzed with a 1 g aliquot in place of the 5 g aliquot, mixed with water. If higher dilutions are required, the methanol extract (medium level) will be analyzed.

10.7.8.8 Surrogate and internal standards are added automatically to all samples at the instrument.

10.7.8.9 The autosampler will heat and stir each sample as it is purged.

10.7.8.10 Soil samples that have low internal standard recovery when analyzed (< 50%) should be reanalyzed once to confirm matrix effect.

10.7.9 Initial Review and Corrective Actions

10.7.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minute 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.

10.7.9.2 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.

Sample internal standard areas are compared to the mid-point of the supplemental initial calibration internal standard areas. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

10.7.9.3 The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

10.7.10 Dilutions

10.7.10.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or 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.

10.7.10.2 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.

10.7.10.3 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.7.11 Instrument Set-up

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. 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.

10.7.12 Recommended Instrument Conditions

10.7.12.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 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:	1-15 mL/minute, dependent upon column specifications

10.7.12.2 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

BFB Analysis

Initial Temperature:	150 ˚C
Initial Hold Time:	0.00 minutes
1st Temperature Program:	50.00 ˚C/minute
Final Temperature:	220 ˚C
Final Time:	4.00 minutes
2nd Temperature Program:	OFF
Post Temperature:	0 ˚C
Post Time:	0.00 minutes
Run Time:	5.40 minutes

Sample Analysis

Initial Temperature:	40 ˚C
Initial Hold Time:	4 minutes
1st Temperature Program:	8 ˚C/minute
Final Temperature:	184 ˚C
2nd Temperature Program:	40 ˚C/minute
Final Temperature:	240 ˚C
Final Hold Time:	2.6 minutes

10.7.12.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 11 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.

10.7.13 Initial Calibration

10.7.13.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and in the public folder *Arizona Calibration Training.*

10.7.13.2 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Certain analytes are prepared at higher concentrations due to poor purge performance. The following calibration curves are maintained. Calibration levels for each analyte are given in the stated tables. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements.

Initial Calibration by Matrix and Method

Method	Matrix	Purge Volume	Calibration Levels
624	Water	5 mL	Table A-3
8260	Water	20 mL	Tables 5 and 5A

Method	Matrix	Purge Volume	Calibration Levels
8260	Soil (low level)	5 mL	Tables 4 and 4A
8260	Soil (Methanol Extract)	5 mL reagent water + 25 µL Methanol	Tables 6 and 6A
Alaska	Soil	See Appendix B	See Appendix B

10.7.13.3 Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points for linear regression and six calibration points for second order calibration. The lowest standard used in the calibration must be at or below the TestAmerica reporting limit.

10.7.13.4 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.

10.7.13.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.7.13.6 Internal standard calibration is used. The internal standards are listed in Tables 7 and 7A. 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 11.4.1, for calculation of response factor.

10.7.13.7 Evaluation of retention times

The relative retention time of each target analyte in each calibration standard should agree within 0.5 min.

10.7.13.8 The % RSD of each of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 13. See Table A-2 for Method 624 criteria.

10.7.13.9 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 12 for the SPCC compounds and required minimum response factors.

10.7.13.10 If the software in use is capable of routinely reporting curve

coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

Note: Additional criteria are stated in the North Carolina QAS.

10.7.13.11 If the software in use is capable of routinely reporting curve coefficients for data, and if the average of all the %RSDs in the calibration is > 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.990.

Note: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

10.7.13.12 Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. All compounds in the ICV must be <35 % drift when compared to the initial calibration, except poor performers (see Table 16) which must be <55% drift. The ICV is generally run at the same concentration as the level 5 standard. See Table A-2 for method 624 criteria.

10.7.13.13 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.7.14.

10.7.13.14 A separate five point calibration must be prepared for analysis of low-level soils. Low-level soils analysis requires the use of a closed vial autosampler. Each standard is prepared by spiking the methanol standard solution through the septum of a VOA vial containing 5 mL of water. 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. Methanol soil extracts should be analyzed using the methanol calibration curve.

10.7.13.15 Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. The primary ion for the single standard must generate a peak clearly visible over background noise (greater than three standard deviations at a minimum) and be free of spectral interferences. 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. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. A footnote or narrative comment should describe the basis of the reported result.

10.7.14 Continuing Calibration

10.7.14.1 The initial calibration must be verified every twelve hours.

10.7.14.2 Continuing calibration begins with analysis of BFB as described in Section 10.7.12.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 4 calibration standard is used as the continuing calibration standard. See Table A-2 for method 624 criteria.

10.7.14.3 The RF data from the standards are compared with the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 11.4.4.

10.7.14.4 The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 12. In addition, the % drift for most non-CCC analytes must be ≤ 35 %, and for poor performers ≤ 50 % (See Table 16), with allowance for up to six target analytes to have a % drift greater than the applicable limit. For agencies that require specific control limits for non-CCC compounds (i.e., State of Arizona) see Table 15. See Table A-2 for method 624 criteria.

Note: Additional criteria are stated in the North Carolina QAS.

10.7.14.4.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 13) must be agreed to with the client.

10.7.14.4.2 Cyclohexanone is unstable in the calibration solution forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists.

10.7.14.5 The retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds when compared to the most recent five-point calibration. The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent five-point calibration.

10.7.14.6 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.7.14.3 and 10.7.14.4, 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.

10.7.14.7 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.)

10.7.14.8 Sodium Bisulfate must be added to the CCV when analyzing samples preserved with it.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and in the public folder, *Arizona Calibration Training*.

11.2 Qualitative Identification

11.2.1 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 or from the NIST Library (same library as used for routine sample analysis). 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.

11.2.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.2.1.2 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.

11.2.1.3 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%.)

11.2.2 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.

11.2.3 All data are subject to two levels of technical review, as described in SOP DV-QA-0020.

11.3 Tentatively Identified Compounds (TICs)

11.3.1 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. The following guidelines apply:

11.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.3.1.2 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%).

11.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.3.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.3.1.5 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 co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

11.3.1.6 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.*

11.4 Calculations.

11.4.1 Response factor (RF):

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$ *Equation 1*

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng
C_x = Concentration of the compound being measured, ng.

11.4.2 Standard deviation (SD):

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{n-1}} \qquad \textit{Equation 2}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of X_i.

11.4.3 Percent relative standard deviation (%RSD):

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \textit{Equation 3}$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

11.4.4 Percent drift between the initial calibration and the continuing calibration:

$$\%Drift = \frac{C_{\text{exp}ected} - C_{found}}{C_{\text{exp}ected}} \times 100\% \qquad \textit{Equation 4}$$

Where:

$C_{expected}$ = *Known concentration in standard.*
C_{found} = *Measured concentration using selected quantitation method.*

11.4.5 See SOP CA-Q-S-005 for more detailed calibration equations.

11.4.6 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 ≤ 15%.

11.4.6.1 Calculation of concentration using Average Response Factors:

$$\text{Concentration } (\mu\text{g/L}) = \frac{x}{\overline{RF}} \qquad \textit{Equation 5}$$

11.4.6.2 Calculation of concentration using Linear fit:

$$\text{Concentration } (\mu\text{g/L}) = A + Bx \qquad \textit{Equation 6}$$

11.4.6.3 Calculation of concentration using Quadratic fit:

$$Concentration(\mu g / L) = A + Bx + Cx^2$$ *Equation 7*

Where:

x = see equations 8, 9, and 10.
A = intercept of the calibration function.
B = slope of calibration function.
C = curvature of a second-order calibration function.

11.4.6.4 Calculation of x for Water and water-miscible waste:

$$x = \frac{A_x I_s D_f}{A_{is} V_0}$$ *Equation 8*

Where:

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).*

A_{is} = *Area of the characteristic ion for the internal standard.*

I_s = *Amount of internal standard added in ng.*

$$D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = *Volume of water purged, mL.*

11.4.6.5 Calculation of x for High-level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)D_f}{(A_{is})(V_a)(W_s)(D)}$$ *Equation 9*

Where:

A_x, I_s, D_f, A_{is}, = *same as used in equation 8 above.*

V_t = *Volume of total extract, mL (typically 25 mL).*

V_a = *Volume of extract added for purging, µL.*

W_s = *Weight of sample extracted, g.*

D = $\frac{100 - \%moisture}{100}$

11.4.6.6 Calculation of x for Low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$ *Equation 10*

Where:

A_x, I_s, D_f, A_{is},	=	*same as used in equation 8 above.*
D	=	*same as in equation 9 above.*
W_s	=	*Weight of sample added to the purge vessel, g.*

11.4.6.7 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x	=	*Area in 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*

In other words, the concentration is equal to x as defined in equations 8, 9, and 10.

11.4.7 MS/MSD Recovery

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100\% \qquad \textit{Equation 11}$$

Where:

SSR	=	*Spike sample result.*
SR	=	*Sample result.*
SA	=	*Spike added.*

11.4.8 Relative % Difference calculation for the MS/MSD:

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}\left(MSR + MSDR\right)} \times 100\% \qquad \textit{Equation 12}$$

Where:

RPD	=	*Relative percent difference.*
MSR	=	*Matrix spike result.*
MSDR	=	*Matrix spike duplicate result.*

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements (e.g., DoD) indicate a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1.1 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 a mid- level calibration.

12.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.1.3 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.

12.2.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

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 procedure, the

policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methanol Waste - Vial Waste and Flammable – Waste Streams A and C

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.3 Acidified Water – Waste Stream W

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996.

15.1.2 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December, 1996.

15.1.3 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December, 1996.

15.1.4 Method 5035A (R1-MIR), Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

15.1.5 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.6 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003..

15.2 40 CFR Part 136, Appendix A (Method 624, Method 603).

15.3 Method AK101 For the Determination of Gasoline Range Organics, Alaska DEC, Version 04/08/02.

16.0 Method Modifications:

Item	Method	Modification
1	SW-846 8260B	Ion 119 is used as the quantitation ion for chlorobenzene-d_5.
2	SW-846 8260B	The quantitation and qualifier ions for some compounds have been

Item	Method	Modification
		changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
3	SW-846 8260B	This SOP has been written to allow for a 20 mL purge volume for waters. An additional 5 mL of DI water is added to all samples, QC and calibration standards. The final purge volume is 25 mL.
4	SW-846 8260B	Method 8260B recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.
5	SW-846 8260B	SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition, most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, if the overall average is $\leq$ 15% then the ICAL is considered acceptable and any compounds that are not $\leq$15% will use linear regression.
6	EPA 624	Method 624 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
7	EPA 624	The tune period for this method is defined as 24 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 24 hour frequency.
8	EPA 624	The initial calibration curve for this method requires at least three points, as shown in Table A-3.
9	EPA 624	Sample concentrations are calculated using the average RRF from the initial calibration curve.
10	EPA 624	Each target analyte is assigned to the closest eluting internal standard.
11	EPA 624	Initial demonstration of Proficiency • The spiking level for the four replicate initial demonstration of proficiency is 20 μg/L. The acceptance criteria are listed in Table A-2
12	EPA 624	Initial calibration curve requirements: • Target compounds must have RSD $\leq$ 35%.

Item	Method	Modification
		• If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.
13	EPA 624	Continuing calibration verification requirements: • The continuing calibration standard is from a different source than the initial calibration standard. The daily CCAL concentration is 20 ug/L. The acceptance criteria are listed in Table A-2. Matrix Spike and LCS Requirements • The matrix spike and LCS are spiked at 20 μg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table A-2.
14	EPA 624	Consistent with the other volatile methods, corrections for recovery are not allowed.
15	EPA 624	Qualitative Identification – The source method states that the relative intensities of ions should agree to within ±20% between the standard and sample spectra. This SOP uses ±30%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)
16	EPA 624	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 OI #10 trap which consists of Tenax/Silica Gel/ Carbon Molecular Sieve or the Supelco Vocarb 3000 which consists of Carbopack B, Carbonxen1000 and 1001.
17	EPA 624	Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
18	EPA 624	The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including chlorobenzene-d_5 and 1,4-dichlorobenzene-d_4, which are not listed in Table 3 of the source method. Toluene-d_8 is used as a surrogate compound, which is also not listed in the source method.
19	EPA 624	The lab is preparing internal standards at 10 ug/L and applying the same criteria designed for 30 ug/L in the Method. The lower concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 ug/L; it provides a higher

Item	Method	Modification
		level of data quality.
20	EPA 624	Method 624 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, we scan from 35 to 300 and include all ions needed for analysis.
21	EPA 624	Method 624 describes dilutions "if response of any m/z" exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.10) is equivalent.
22	EPA 624	Method 624 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta- and para-xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. meta- and para-xylenes are reported as an isomeric pair. Any development work to add compounds would take this into account.
23	624	The source method recommends Method 603 as the preferred method for Acrolein and Acrylonitrile. Method 624 is recommended as a screening method (see section 1.2 of Methods 603 and 624). Calibration and quality control samples indicate that the conditions described in this SOP are suitable for the analysis of Acrolein and Acrylonitrile. EPA's Method Update Rule (MUR), May 18, 2012, allows the addition of acrolein and acrylonitrile to Method 624, using the preservation, holding time and QC acceptance criteria from Method 603. As states implement the MUR Method 624 becomes a determinative method for these two analytes. Until such time, Method 624 remains a screening method for regulatory compliance.
24	SW846 5035	The source method recommends adding approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~ 1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. TestAmerica Denver does not recommend the use of sodium bisulfate to preserve soil samples, but encourages clients to collect samples using other available methods. The use of this preservative has been shown to cause difficulties recovering more reactive analytes on the purge and trap system (e.g. 2-Chloroethyl vinyl ether, acrylamide).

17.0 Attachments

Table 1. TestAmerica Primary List Reporting Limits for 8260B
Table 2. TestAmerica 8260 Secondary List Reporting Limits
Table 3. TestAmerica Appendix IX List Reporting Limits
Table 4. TestAmerica Non-Standard Compound List Reporting Limits
Table 5. Soil Calibration Levels, 5-gram Purge (μg/Kg)

	(Standard Mixes: MV-Main & MV-Main GasKe)
Table 5A.	Soil Calibration Levels, 5 gram Purge (µg/Kg) (Standards: MV-Supp Std and MV-2 Cleve)
Table 6.	Water 8260 List Calibration Levels (µg/L) (Standards: MV-Main and MV-Main GasKe)
Table 6A	Water 8260 List Calibration Levels (µg/L) (Standards: MV-Supp Std and MV- 2 Cleve)
Table 7.	Medium Level Soil 8260 List Calibration Levels (µg/Kg) (Standards: MV-Main and MV-Main GasKe)
Table 7A.	Medium Level Soil 8260 List Calibration Levels (µg/Kg) (Standards: MV-Supp Std and MV- 2 Cleve)
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Table 8A.	Automatically Added Internal Standards
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Table 10.	Matrix Spike and LCS Standard
Table 11.	BFB Key Ion Abundance Criteria
Table 12.	SPCC Compounds and Minimum Response Factors
Table 13.	CCC Compounds
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Table 15.	State of Arizona ICV/CCV Quality Control Limits
Table 16.	List 1 Poorly performing Compounds
Table A-1.	Method 624 Analytes and Reporting Limits, 5-mL Purge
Table A-2.	Method 624 QC Acceptance Criteria
Table A-3.	Calibration Levels for 624, 5 mL Purge
Appendix A.	Modifications for Analysis of 1,4-Dioxane, 1,2,3-Ttrichloropropane, 1,2-Dibromo-3-chloropropane, and 1,2-Dibromoethane by Selected Ion Monitoring
Table Ap-1.	TAL Method 8260SIM Standard Reporting Limits
Table Ap-2.	Method 8260SIM Calibration Levels
Table Ap-3.	Method 8260SIM LCS Spike Concentrations
Table Ap-4.	8260SIM Surrogate Compounds
Table Ap-5.	8260SIM Internal Standard Compounds
Table Ap-6.	8260 Selected Masses
Table Ap-7.	Suggested Instrument Conditions for 8260SIM
Appendix B	Modifications for Analysis of Soils Collected for the State of Alaska
Table Bp-1:	TestAmerica 8260 Reporting Limits – AK Soils
Table Bp-2:	Calibration Levels for 8260, 5035FM_AK
Table Bp-3:	5035FM_AK Calibration Levels (µg/Kg) (Standards: MV-Supp Std and MV-2 Cleve)
Attachment 1.	Gas Standards Tracking Log

18.0 Changes from Previous Revision

- Revision 9, dated 04 January 2013
  - Added section 9.8 to address the 2012 MUR QC requirements
- Revision 8, dated 28 September 2012
  - Added to compounds to the reporting limit, characteristic ion and calibration tables

to match TALS.

- Revision 7, dated 27 July 2012
  - Added sodium bisulfate to Section 7.
  - Revised Section 8 to include Terra Core samplers and moved instructions on sample preparation and handling in the lab to Section 10. Reorganized sampling and preservation information into tables. Updated information including footnote on Holding Time and preservation table for water regarding Method Update Rule that approves use of Method 624 for analysis of acrolein and acrylonitrile.
  - Removed flowcharts from Section 8.
  - Revised Section 9.1
  - Revised Section 10.
  - Updated reference section to include Method 603, Method 5035A, and Method 8000B and 8000C.
  - Revised Method Modifications #23
  - Updated tables to reflect current practice.
  - Added Appendix B for the analysis of soils using the AK methanol extraction procedure.
  - Formatting and editorial changes throughout

- Revision 6.4, dated 28 December 2011
  - Changed the column ID and film thickness in section 6.1.8.1
  - Updated the calibration levels in Table AP-2

- Revision 6.3, dated 26 October 2011
  - Added Section 4.6 regarding interferences with toluene-d_8 surrogate when potassium permanganate may have been added to sample
  - Updated path to QAS folders in the public folders, section 9.7
  - Added J. T. Baker Antifoam B and reagent sand, sections 7.3, 7.4
  - Added description of procedure for use of antifoaming agent B, section 10.1.3.8
  - Formatting

- Revision 6.2, dated 25 August, 2011
  - Added requirements to section 9.4 for the use of Ottawa sand in soil LCS's.

- Revision 6.1, dated 31 January, 2011
  - Added details to Appendix A for the analysis of soils by SIM
  - Added Tables AP-1 through Ap-7
  - Added Attachment 1, Gas Standards Tracking Log
  - Added section 11.1 referencing corporate SOP CA-Q-S-005 "Calibration Curves"

- Revision 6, dated 02 November, 2010
  - Added analysis information concerning BFB

- Revision 4, dated May 5, 2010
  - Updated Tables to reflect current report limits.
  - Updated low level procedure to include water option for preservation.
  - Updated surrogate and spike amounts.

- Revision 3.1, dated 11 December 2009
  - Added Trichloroethene to Table 12.
  - Updated section 16 to describe the process of adding and additional 5 mL of DI

water to all samples and QC.
  - Added a note to section 9.4 that marginal exceedances are not allowed for some programs.
  - Updated the language in section 16 item 5 to describe the current practice.

- Revision 3.0, dated 21 January 2009
  - Added clarification of sample preservation requirements to section 8.
  - Adjusted Table 16 for South Carolina requirements to utilize default limits.
  - Added Table 8A for AFCEE water calibration levels.

- Revision 2.1, dated 16 July 2007
  - Add reference to North Carolina QAS for additional requirements to sections 9.6, 10.4.8, and 10.5.4.
  - Remove Nitrogen as an allowable substitution for Helium in section 6.8.
  - Added the current list of spike compounds to Table 12.
  - Updated references to include 5030B and 5035.
  - Removed EPA 524.2 references.

- Revision 2.0
  - The method blank acceptance criteria and corrective actions were updated in Section 9.4.

Table 1. TestAmerica Primary List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	2	10	500
Chloromethane	74-87-3	2	10	500
Bromomethane	74-83-9	2	10	500
Vinyl chloride	75-01-4	1	5	500
Chloroethane	75-00-3	2	10	500
Trichlorofluoromethane	75-69-4	2	10	500
Acrolein	107-02-8	20	50	5,000
Acetone	67-64-1	10	20	1,000
Trichlorotrifluoroethane	76-13-1	3	20	1,000
Ethanol	64-17-5	300	600	10,000
Iodomethane	74-88-4	1	5	250
Carbon disulfide	75-15-0	2	5	250
Methylene chloride	75-09-2	2	5	250
tert-Butyl alcohol	75-65-0	50	200	10,000
1,1-Dichloroethene	75-35-4	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	2.5	125
Acrylonitrile	107-13-1	20	50	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	20	250
Hexane	110-54-3	2	5	250
cis-1,2-Dichloroethene	156-59-2	1	2.5	125
1,2-Dichloroethene (Total)	540-59-0	1	5	250
Tetrahydrofuran	109-99-9	7	20	1,000
Chloroform	67-66-3	1	10	250
1,2-Dichloroethane	107-06-2	1	5	250
Dibromomethane	74-95-3	1	5	250
2-Butanone	78-93-3	6	20	1,000
1,4-Dioxane	123-91-1	200	500	25,000
1,1,1-Trichloroethane	71-55-6	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250

Table 1. TestAmerica Primary List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
cis-1,3-Dichloropropene	10061-01-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Dibromochloromethane	124-48-1	1	5	250
1,2-Dibromoethane	106-93-4	1	5	250
1,2,3-Trichloropropane	96-18-4	2.5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Benzene	71-43-2	1	5	250
Ethylmethacrylate	97-63-2	3	5	250
trans-1,3-Dichloropropene	10061-02-6	3	5	250
Bromoform	75-25-2	1	5	250
4-Methyl-2-pentanone	108-10-1	5	20	1,000
2-Hexanone	591-78-6	5	20	1,000
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
2-Chloroethyl vinyl ether[2]	110-75-8	N/A[2]	50	2,500
Vinyl acetate	108-05-4	3	10	500
Chlorobenzene	108-90-7	1	5	250
Ethylbenzene	100-41-4	1	5	250
Styrene	100-42-5	1	5	250
trans-1,4-Dichloro-2-butene	110-57-6	3	5	250
m- and p-Xylenes	179601-23-1	2	3.5	250
o-xylene	95-47-6	1	2.5	125
Total xylenes	1330-20-7	2	10	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
1,2-Dichlorobenzene	95-50-1	1	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.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2. TestAmerica 8260 Secondary List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
2,2-Dichloropropane	590-20-7	1	5	250
Bromochloromethane	74-97-5	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
1,3-Dichloropropane	142-28-9	1	5	250
1-Chlorohexane	544-10-5	1	5	500
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
Isopropylbenzene	98-82-8	1	5	250
Bromobenzene	108-86-1	1	5	250
n-Propylbenzene	103-65-1	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	250
sec-Butylbenzene	135-98-8	1	5	250
4-Isopropyltoluene	99-87-6	1	5	250
n-Butylbenzene	104-51-8	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Naphthalene	91-20-3	1	5	500
Hexachlorobutadiene	87-68-3	1	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	250
2-Pentanone	107-87-9	5	10	500
cis-1,4-Dichloro-2-butene	1476-11-5	3	5	250
Ethylene oxide	75-21-8	600	3,000	150,000

Table 3. TestAmerica Appendix IX List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
Allyl Chloride	107-05-1	2	10	500
Acetonitrile	75-05-8	30	100	5,000
Dichlorofluoromethane	75-43-4	2	10	25,000
Isopropyl ether	108-20-3	10	50	2,500
Chloroprene	126-99-8	1	5	500
n-Butanol	71-36-3	60	200	10,000
Propionitrile	107-12-0	20	50	1,000
Methacrylonitrile	126-98-7	10	50	2,500
Isobutanol	78-83-1	110	200	10,000
Methyl methacrylate	80-62-6	4	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	10	500
Ethyl ether	60-29-7	2	10	500
Ethyl Acetate	141-78-6	5	10	500
2-Nitropropane	79-46-9	5	10	500
Cyclohexanone[2]	108-94-1	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	1	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.

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4. TestAmerica Non-Standard List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
1,1,1-Trifluoro-2,2-Dichloroethane	306.83-2	2.0	5.0	1000
1,2,3-Trimethylbenzene	526-73-8	2.0	5.0	250
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	2.0	5.0	250
1,2-Dichloro-1,1,2-Trifluoroethane	354-23-4	2.0	5.0	250
1,3,5-Trichlorobenzene	108-70-3	5.00	***	***
2,2,3-Trimethylbutane	464-06-2	5.00	***	***
2,2-Dimethylpentane	590-35-2	5.00	***	***
2,3-Dimethylpentane	565-59-3	5.00	***	***
2,4-Dimethylpentane	108-08-7	5.00	***	***
2-Chloro-1,1,1-Trifluoroetane	75-88-7	5.00	5.0	250
2-Methylhexane	591-76-4	5.00	***	***
3,3-Dimethylpentane	562-49-2	5.00	***	***
3-Ethylpentane	617-78-7	5.00	***	***
3-Methylhexane	589-34-4	5.00	***	***
Chlorotrifluoroethene	79-38-9	5.00	5.0	250
Cyclohexane	110-82-7	2.0	5.0	250
Dimethyl Disulfide	624-92-0	5.00	***	***
Isopropyl Alcohol	67-63-0	40	200	10,000
Methyl Acetate	79-20-9	5.0	10	1000
Methylcyclohexane	108-87-2	1.0	5.0	250
n-Heptane	142-82-5	5.00	***	***
n-Nonyl Aldehyde	124-19-6	10.00	***	***
Propene Oxide	75-56-9	50	3000	250
Sec-Butyl Alcohol	78-92-2	***	200	***
Tert-amyl methyl ether	994-05-8	5	5.0	1000
Tert-butyl ethyl ether	637-92-3	5	5.0	1000
Tetrahydrothiophene	110-01-0	2.0	5.0	***

[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 5. Soil Calibration Levels, 5-gram Purge[1] (Standard Mixes: MV-Main & MV-Main GasKe)								
Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,1-Trichloroethane	1	2	5	10	20	50	100	200
1,1,2,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,2-Trichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethene	1	2	5	10	20	50	100	200
1,1-Dichloropropene	1	2	5	10	20	50	100	200
1,2,3-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,3-Trichloropropane	1	2	5	10	20	50	100	200
1,2,4-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,4-Trimethylbenzene	1	2	5	10	20	50	100	200
1,2-Dibromo-3-chloropropane	1	2	5	10	20	50	100	200
1,2-Dichlorobenzene	1	2	5	10	20	50	100	200
1,2-Dichloroethane	1	2	5	10	20	50	100	200
1,2-Dichloropropane	1	2	5	10	20	50	100	200
1,3,5-Trimethylbenzene	1	2	5	10	20	50	100	200
1,3-Dichlorobenzene	1	2	5	10	20	50	100	200
1,3-Dichloropropane	1	2	5	10	20	50	100	200
1,4-Dichlorobenzene	1	2	5	10	20	50	100	200
1,4-Dioxane	50	100	250	500	1,000	2,500	5,000	10,000
1-Chlorohexane	1	2	5	10	20	50	100	200
2,2-Dichloropropane	1	2	5	10	20	50	100	200
2-Butanone	4	8	20	40	80	200	400	800
2-Chloro-1,3-butadiene	1	2	5	10	20	50	100	200
2-Chlorotoluene	1	2	5	10	20	50	100	200
2-Hexanone	4	8	20	40	80	200	400	800
4-Chlorotoluene	1	2	5	10	20	50	100	200
4-Isopropyltoluene	1	2	5	10	20	50	100	200
4-Methyl-2-pentanone	4	8	20	40	80	200	400	800
Acetone	4	8	20	40	80	200	400	800
Acetonitrile	4	8	20	40	80	200	400	800
Acrolein	4	8	20	40	80	200	400	800
Acrylonitrile	4	2	5	10	20	50	100	200

Table 5. Soil Calibration Levels, 5-gram Purge[1]
(Standard Mixes: MV-Main & MV-Main GasKe)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Benzene	1	2	5	10	20	50	100	200
Bromobenzene	1	2	5	10	20	50	100	200
Bromoform	1	2	5	10	20	50	100	200
Bromomethane	1	2	5	10	20	50	100	200
Carbon tetrachloride	1	2	5	10	20	50	100	200
Chlorobenzene	1	2	5	10	20	50	100	200
Chlorobromomethane	1	2	5	10	20	50	100	200
Chloroethane	1	2	5	10	20	50	100	200
Chloroform	1	2	5	10	20	50	100	200
Chloromethane	1	2	5	10	20	50	100	200
cis-1,2-Dichloroethene	1	2	5	10	20	50	100	200
cis-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Cyclohexanone	40	80	200	400	800	2,000	4,000	8,000
Chlorodibromomethane	1	2	5	10	20	50	100	200
Dibromomethane	1	2	5	10	20	50	100	200
Dichlorobromomethane	1	2	5	10	20	50	100	200
Dichlorodifluoromethane	1	2	5	10	20	50	100	200
Ethanol	50	100	250	500	1,000	2,500	5,000	10,000
Ethylbenzene	1	2	5	10	20	50	100	200
Ethylene dibromide	1	2	5	10	20	50	100	200
Hexachlorobutadiene	1	2	5	10	20	50	100	200
Iodomethane	1	2	5	10	20	50	100	200
Isobutyl alcohol	20	40	100	200	400	1,000	2,000	4,000
Isopropyl ether	5	10	25	50	100	250	500	1,000
Isopropylbenzene	1	2	5	10	20	50	100	200
m- and p-Xylenes	2	4	10	20	40	100	200	400
Methacrylonitrile	10	20	50	100	200	500	1,000	2,000
Methylene chloride	1	2	5	10	20	50	100	200
Naphthalene	1	2	5	10	20	50	100	200
n-Butanol	30	60	150	300	600	1,500	3,000	--
n-Butylbenzene	1	2	5	10	20	50	100	200
n-Propylbenzene	1	2	5	10	20	50	100	200
o-Xylene	1	2	5	10	20	50	100	200
Propionitrile	10	20	50	100	200	500	1,000	2,000

Table 5. Soil Calibration Levels, 5-gram Purge[1] (Standard Mixes: MV-Main & MV-Main GasKe)								
Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
sec-Butylbenzene	1	2	5	10	20	50	100	200
Styrene	1	2	5	10	20	50	100	200
2-Methyl-2-propanol (tert-Butyl alcohol)	20	40	100	200	400	1,000	2,000	4,000
tert-Butylbenzene	1	2	5	10	20	50	100	200
Tetrachloroethene	1	2	5	10	20	50	100	200
Toluene	1	2	5	10	20	50	100	200
trans-1,2-Dichloroethene	1	2	5	10	20	50	100	200
trans-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Trichloroethene	1	2	5	10	20	50	100	200
Trichlorofluoroethane	1	2	5	10	20	50	100	200
Vinyl chloride	1	2	5	10	20	50	100	200

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 5A: Soil Calibration Levels, 5-gram Purge, µg/Kg[1] (Standards: MV-Supp Std and MV-2 Cleve)							
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloro-ethane	2	5	10	20	50	100	200
1,1,2-Trichloro-1,2,2-trifluoroethane	2	5	10	20	50	100	200
1,2,3-Trimethylbenzene	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2,2-tetrafluroethane	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2-trifluoroethane	2	5	10	20	50	100	200
2-Chloroethyl vinyl ether	2	5	10	20	50	100	200
2-Nitropropane	2	5	10	20	50	100	200
2-Pentanone	8	20	40	80	200	400	800
3-Chloro-1-propene (Allyl Chloride)	2	5	10	20	50	100	200
Carbon disulfide	2	5	10	20	50	100	200
cis-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Cyclohexane	2	5	10	20	50	100	200

Table 5A: Soil Calibration Levels, 5-gram Purge, µg/Kg[1] (Standards: MV-Supp Std and MV-2 Cleve)							
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Dichlorofluoromethane	2	5	10	20	50	100	200
Ethyl Acetate	4	10	20	40	100	200	400
Ethyl ether	2	5	10	20	50	100	200
Ethyl methacrylate	4	10	20	40	100	200	400
Ethylene Oxide	250	625	1,250	2,500	6,250	12,500	25,000
Hexane	2	5	10	20	50	100	200
Isopropyl alcohol	40	100	200	400	1000	2000	4,000
Methyl acetate	10	25	50	100	250	500	1,000
Methyl methacrylate	4	10	20	40	100	200	400
Methyl *tert*-butyl ether (MTBE)	2	5	10	20	50	100	200
Methylcyclohexane	2	5	10	20	50	100	200
sec-Butyl alcohol	60	150	300	600	1500	3000	6,000
tert-Amyl methyl ether	10	25	50	100	250	500	1,000
tert-Butyl ethyl ether	10	25	50	100	250	500	1,000
Tetrahydrofuran	4	10	20	40	100	200	400
trans-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Trichlorofluoromethane	2	5	10	20	50	100	200
Vinyl acetate	4	10	20	40	100	200	400

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,4-Dioxane	15	50	100	250	500	1,500	3,000
1-Chlorohexane	0.3	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	1.2	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.3	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
2-Hexanone	1.2	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	6	20	40	100	200	600	1,200
4-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.3	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	1.2	4.0	8.0	20	40	120	240
Acetone	1.2	4.0	8.0	20	40	120	240
Acetonitrile	3	10	20	50	100	300	600
Acrolein	3	10	20	50	100	300	600
Acrylonitrile	3	10	20	50	100	300	600

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.3	1.0	2.0	5.0	10	30	60
Bromobenzene	0.3	1.0	2.0	5.0	10	30	60
Bromoform	0.3	1.0	2.0	5.0	10	30	60
Bromomethane	0.3	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.3	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.3	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.3	1.0	2.0	5.0	10	30	60
Chloroethane	0.3	1.0	2.0	5.0	10	30	60
Chloroform	0.3	1.0	2.0	5.0	10	30	60
Chloromethane	0.3	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Cyclohexanone	12	40	80	200	400	1,200	2,400
Dibromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.3	1.0	2.0	5.0	10	30	60
Ethanol	15	50	100	250	500	1,500	3,000
Ethylbenzene	0.3	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.3	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.3	1.0	2.0	5.0	10	30	60
Iodomethane	0.3	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	6	20	40	100	200	600	1,200
Isopropyl ether	1.5	5.0	10	25	50	150	300
Isopropylbenzene	0.3	1.0	2.0	5.0	10	30	60
m and p Xylenes	0.6	2.0	4.0	10	20	60	120
Methacrylonitrile	3	10	20	50	100	300	600
Methylene chloride	0.3	1.0	2.0	5.0	10	30	60
Naphthalene	0.3	1.0	2.0	5.0	10	30	60
n-Butanol	9.0	30	60	150	300	900	1,800
n-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.3	1.0	2.0	5.0	10	30	60
o-Xylene	0.3	1.0	2.0	5.0	10	30	60
Propionitrile	3.0	10	20	50	100	300	600
sec-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.3	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.3	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.3	1.0	2.0	5.0	10	30	60
Toluene	0.3	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Trichloroethene	0.3	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.3	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.3	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6A: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1-Trifluoro-2,2-dichloroethane	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	1.0	2.0	5.0	10	30	60
2-Nitropropane	1.0	2.0	5.0	10	30	60
2-Pentanone	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	1.0	2.0	5.0	10	30	60
Carbon disulfide	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Cyclohexane	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	1.0	2.0	5.0	10	30	60
Ethyl acetate	2.0	4.0	10	20	60	120
Ethyl ether	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	2.0	4.0	10	20	60	120

Table 6A: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Ethylene oxide	125	250	625	1,250	3,750	7,500
Hexane	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	20	40	100	200	600	1,200
Methyl acetate	5.0	10	25	50	150	300
Methylcylcohexane	1.0	2.0	5.0	10	30	60
Methyl methacrylate	2.0	4.0	8.0	20	60	120
Methyl *tert*-butyl ether (MTBE)	1.0	2.0	5.0	10	30	60
Propene oxide	20	100	250	500	1,500	3,000
sec-Butyl alcohol	30	60	150	300	900	1,800
tert-Amyl methyl ether	5.0	10	25	50	150	300
tert-Butyl ethyl ether	5.0	10	25	50	150	300
Tetrahydrofuran	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Vinyl acetate	2.0	4.0	10	20	60	120

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,4-Dioxane	25	50	100	250	500	1,500	3,000
1-Chlorohexane	0.5	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	2.0	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.5	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
2-Hexanone	2.0	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	40	100	200	600	1,200
4-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.5	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	2.0	4.0	8.0	20	40	120	240
Acetone	2.0	4.0	8.0	20	40	120	240
Acetonitrile	5	10	20	50	100	300	600
Acrolein	5	10	20	50	100	300	600
Acrylonitrile	5	10	20	50	100	300	600

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.5	1.0	2.0	5.0	10	30	60
Bromobenzene	0.5	1.0	2.0	5.0	10	30	60
Bromoform	0.5	1.0	2.0	5.0	10	30	60
Bromomethane	0.5	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	30	60
Chloroethane	0.5	1.0	2.0	5.0	10	30	60
Chloroform	0.5	1.0	2.0	5.0	10	30	60
Chloromethane	0.5	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexanone	20	40	80	200	400	1,200	2,400
Dibromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethanol	25	50	100	250	500	1,500	3,000
Ethylbenzene	0.5	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.5	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	30	60
Iodomethane	0.5	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	10	20	40	100	200	600	1,200
Isopropyl ether	2.5	5.0	10	25	50	150	300
Isopropylbenzene	0.5	1.0	2.0	5.0	10	30	60
m and p Xylenes	1.0	2.0	4.0	10	20	60	120
Methacrylonitrile	5	10	20	50	100	300	600
Methylene chloride	0.5	1.0	2.0	5.0	10	30	60
Naphthalene	0.5	1.0	2.0	5.0	10	30	60
n-Butanol	15	30	60	150	300	900	1,800
n-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.5	1.0	2.0	5.0	10	30	60
o-Xylene	0.5	1.0	2.0	5.0	10	30	60
Propionitrile	5.0	10	20	50	100	300	600
sec-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.5	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.5	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.5	1.0	2.0	5.0	10	30	60
Toluene	0.5	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Trichloroethene	0.5	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.5	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 7A: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	0.5	1.0	2.0	5.0	10	30	60
2-Nitropropane	0.5	1.0	2.0	5.0	10	30	60
2-Pentanone	2.0	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	0.5	1.0	2.0	5.0	10	30	60
Carbon disulfide	0.5	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexane	0.5	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethyl acetate	1.0	2.0	4.0	10	20	60	120
Ethyl ether	0.5	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	1.0	2.0	4.0	10	20	60	120

Table 7A: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Ethylene oxide	62.5	125	250	625	1,250	3,750	7,500
Hexane	0.5	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	10	20	40	100	200	600	1,200
Methyl acetate	2.5	5.0	10	25	50	150	300
Methylcylcohexane	0.5	1.0	2.0	5.0	10	30	60
Methyl methacrylate	1.0	2.0	4.0	8.0	20	60	120
Methyl *tert*-butyl ether (MTBE)	0.5	1.0	2.0	5.0	10	30	60
Propene oxide	10	20	100	250	500	1,500	3,000
sec-Butyl alcohol	15	30	60	150	300	900	1,800
tert-Amyl methyl ether	2.5	5.0	10	25	50	150	300
tert-Butyl ethyl ether	2.5	5.0	10	25	50	150	300
Tetrahydrofuran	1.0	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Vinyl acetate	1.0	2.0	4.0	10	20	60	120

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 8. Manually Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	20	96
Chlorobenzene-d_5	20	119
1,4-Dichlorobenzene-d_4	20	152

NOTES:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

Table 8A. Automatically Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	250	96
Chlorobenzene-d_5	250	119
1,4-Dichlorobenzene-d_4	250	152

NOTES:

1) 1 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the internal standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added internal standard concentrations.

Table 9. Manually Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	20
Dibromofluoromethane	20
Toluene-d_8	20
4-Bromofluorobenzene	20

NOTES:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-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 9A. Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	250
Dibromofluoromethane	250
Toluene-d_8	250
4-Bromofluorobenzene	250

NOTES:

1) 1 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the surrogate standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added surrogate standard concentrations.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10. Matrix Spike and LCS Standard

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	40
Methylene Chloride	40
Trans-1,2-Dichloroethene	40
1,1-Dichloroethane	40
1111-Trichloroethane	40
Carbon Tetrachloride	40
Benzene	40
Trichloroethene	40
1,2-Dichloropropane	40
Bromodichloromethane	40
Toluene	40
Tetrachloroethene	40
Chlorobenzene	40
Ethylbenzene	40
1,4-Dichlorobenzene	40
1,3-Dichlorobenzene	40

NOTES:

1) 2.5 µL of the standard is added to the LCS or matrix spike sample. This results in a concentration of each spike analyte in the sample of 5 µg/L for a 20 mL purge.
2) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.
3) Full analyte spikes or different compounds may also be used at the laboratory's option or at client request.

Table 11. 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 12. 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 13. 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 14. Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	--
Dichlorodifluoromethane	85	87	50, 101,103
Dibromofluoromethane	111	113	--
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
Ethanol	45	46	--
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
2-Butanone	43	72**	--
Tetrahydrofuran	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

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Benzene	78	52	77
Trichloroethene	95	130***	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	41	76	78
Acetonitrile	41	40	--
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

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
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	--
2,2-Dichloropropane	77	97	--
Bromochloromethane	128	49	130
1,1-Dichloropropene	75	39	110
1,3-Dichloropropane	76	41	78
1-Chlorohexane	91	55	41
1,1,1,2-Tetrachloroethane	131	133	--
Bromobenzene	156	158	77
n-Propylbenzene	120	91	65
2-Chlorotoluene	126	91	65
1,3,5-Trimethylbenzene	105	120	77
4-Chlorobenzene	126	91	89
t-Butylbenzene	119	134	91
sec-Butylbenzene	134	105	--
4-Isopropyltoluene	119	134	91
n-Butylbenzene	91	92	134
1,2,4-Trichlorobenzene	180	182	--
Hexachlorobutadiene	225	227	223
Naphthalene	128	127	--
1,2,3-Trichlorobenzene	180	182	--
1,1,1-Trifluoro-2,2-Dichloroethane	83	133	--
1,2,3-Trimethylbenzene	105	120	91
1,2,4-Trimethylbenzene	105	120	119
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	85	87	--
1,2-Dichloro-1,1,2-Trifluoroethane	117	67	85
1,3,5-Trichlorobenzene	180	182	184
2,2,3-Trimethylbutane	57	43	85
2,2-Dimethylpentane	57	43	85
2,3-Dimethylpentane	56	71	73

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
2,4-Dimethylpentane	43	57	85
2-Chloro-1,1,1-Trifluoroetane	118	83	69
2-Methylhexane	43	85	57
3,3-Dimethylpentane	43	71	--
3-Ethylpentane	43	70	71
3-Methylhexane	43	57	71
4-Chlorotoluene	126	91	89
2-Pentanone	43	86	--
Chlorotrifluoroethene	116	66	97
Cis-1,4-Dichloro-2-butene	53	75	89
Cyclohexane	56	84	55
Dimethyl Disulfide	94	79	45
Ethylene Dibromide	107	109	--
Ethylene Oxide	43	44	--
Isopropyl Alcohol	45	43	--
Methyl Acetate	43	74	59
Methylcyclohexane	55	83	41
m-Xylene & p-Xylene	91	106	77
n-Heptane	43	100	71
n-Nonyl Aldehyde	46	44	207
O-Xylene	106	91	--
Propene Oxide	58	43	57
Sec-Butyl Alcohol	45	59	--
Tert-amyl methyl ether	73	55	87
Tert-butyl ethyl ether	59	87	57
Tetrahydrothiophene	60	88	45

* 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.

*** Used as quantitation ion for method 624.

Table 15. State of Arizona ICV/CCV Quality Control Limits

QC Limits not specified in method	Default QC (method specified or laboratory historical if not specified)
CCV Non-CCC compounds	CCC limits (≤30%)
ICV	Same as CCV (≤30%)
Reporting Limit	Must be supported by low level initial calibration standard
LCS/LCSD	Lab historical
MS/MSD	Lab historical

NOTES:

1) Based on ADHS Rule A.A.C.R9-14-615.C.8. Director approved on June 29, 2005 for the labs to use default limits as an alternative to developing statistically derived limits.

Table 16. List 1 Poorly Performing Compounds

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer."

Acetone	1,2-Dichloro-1,1,2,2-tetrafluoroethane
Acetonitrile	Ethanol
Acrolein	Ethyl acetate
Acrylonitrile	Ethylene oxide
n-Butanol	2-Hexanone
2-Butanone (MEK)	Isobutyl alcohol
tert-Butyl alcohol	Isopropanol
Carbon disulfide	Methacrylonitrile
2-Chloroethyl vinyl ether	Methyl acetate
2-Chloro-1,1,1-trifluoroethane	4-Methyl-2-pentanone
Chlorotrifluoroethene	2-Nitropropane
cis-1,4-Dichloro-2-butene	2-Pentanone
trans-1,4-Dichloro-2-butene	2-Propanol
Dichlorodifluoromethane	Propionitrile
Dichlorofluoromethane	Tetrahydrofuran
1,2-Dibromo-3-chloropropane (DBCP)	Tetrahydrothiophene
1,2-Dichlorotetrafluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
1,2-Dichloro-1,1,2-trifluoroethane (Freon 123a)	Trichlorofluoromethane
2,2-Dichloro-1,1,1-trifluoroethane	Vinyl acetate
1,4-Dioxane	

Table A-1. Method 624 Analytes and Reporting Limits, 5-mL Purge

Analytes	µg/L
Acrolein[1]	100
Acrylonitrile[1]	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	10
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	15
Vinyl chloride	10

[1] Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis.

Table A-2. Method 624 QC Acceptance Criteria

Analytes[1]	Daily QC Check (CCV) Acceptance Criteria (20 µg/L spike)	Mean Recovery, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Std Dev, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Matrix Spike and LCS Acceptance Criteria (% Recovery)
Acrolein[2]	45.9-54.1	42.9-60.1	4.6	88-118
Acrylonitrile[2]	41.2-58.8	33.1-66.9	9.9	71-135
Benzene	12.8 - 27.2	15.2 - 26.0	6.9	37 - 151
Bromodichloromethane	13.1 - 26.9	10.1 - 28.0	6.4	35 - 155
Bromoform	14.2 - 25.8	11.4 - 31.1	5.4	45 - 169
Bromomethane	2.8 - 37.2	D - 41.2	17.9	D - 242
Carbon tetrachloride	14.6 - 25.4	17.2 - 23.5	5.2	70 - 140
Chlorobenzene	13.2 - 26.8	16.4 - 27.4	6.3	37 - 160
Chloroethane	7.6 - 32.4	8.4 - 40.4	11.4	14 - 230
2-Chloroethyl vinyl ether	D - 44.8	D - 50.4	25.9	D - 305
Chloroform	13.5 - 26.5	13.7 - 24.2	6.1	51 - 138
Chloromethane	D - 40.8	D - 45.9	19.8	D - 273
Dibromochloromethane	13.5 - 26.5	13.8 - 26.6	6.1	53 - 149
1,2-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,3-Dichlorobenzene	14.6 - 25.4	17.0 - 28.8	5.5	59 - 156
1,4-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,1-Dichloroethane	14.5 - 25.5	14.2 - 28.5	5.1	59 - 155
1,2-Dichloroethane	13.6 - 26.4	14.3 - 27.4	6.0	49 - 155
1,1-Dichloroethene	10.1 - 29.9	3.7 - 42.3	9.1	D - 234
trans-1,2-Dichloroethene	13.9 - 26.1	13.6 - 28.5	5.7	54 - 156
1,2-Dichloropropane	6.8 - 33.2	3.8 - 36.2	13.8	D - 210
cis-1,3-Dichloropropene	4.8 - 35.2	1.0 - 39.0	15.8	D- 227
trans-1,3-Dichloropropene	10.0 - 30.0	7.6 - 32.4	10.4	17- 183
Ethylbenzene	11.8 - 28.2	17.4 - 26.7	7.5	37 - 162
Methylene chloride	12.1 - 27.9	D - 41.0	7.4	D - 221
1,1,2,2-Tetrachloroethane	12.1 - 27.9	13.5 - 27.2	7.4	46 - 157
Tetrachloroethene	14.7 - 25.3	17.0 - 26.6	5.0	64 - 148
Toluene	14.9 - 25.1	16.6 - 26.7	4.8	47 - 150
1,1,1-Trichloroethane	15.0 - 25.0	13.7 - 30.1	4.6	52 - 162
1,1,2-Trichloroethane	14.2 - 25.8	14.3 - 27.1	5.5	52 - 150
Trichloroethene	13.3 - 26.7	18.6 - 27.6	6.6	71 - 157
Trichlorofluoromethane	9.6 – 30.4	8.9- 31.5	10.0	17 - 181
Vinyl chloride	0.8 – 39.2	D - 43.5	20.0	D - 251

[1] Analytes not listed on the table must meet a CCV drift criteria of ± 30%. Method 624 does not specify second source (ICV) criteria. The laboratory has adopted criteria of ± 30% difference for the ICV. The LIMS requires a minimum value of 10% for the lower limit when D is noted in the reference table.

[2] Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis. Per the MUR, QC criteria from Method 603 are to be applied and are presented here.

Table A-3. Calibration Levels for 624, 5 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Acetone	20	40	80	200	400
Acrolein	50	100	200	500	1000
Acrylonitrile	50	100	200	500	1000
Benzene	5.0	10	20	50	100
Bromoform	5.0	10	20	50	100
Bromomethane	5.0	10	20	50	100
2-Butanone	20	40	80	200	400
Carbon disulfide	5.0	10	20	50	100
Carbon tetrachloride	5.0	10	20	50	100
Chlorobenzene	5.0	10	20	50	100
Chlorodibromomethane	5.0	10	20	50	100
Chloroethane	5.0	10	20	50	100
2-Chloroethyl vinyl ether	5.0	10	20	50	100
Chloroform	5.0	10	20	50	100
Chloromethane	5.0	10	20	50	100
1,2-Dibromo-3-chloropropane	5.0	10	20	50	100
1,2-Dibromoethane (EDB)	5.0	10	20	50	100
Dibromomethane	5.0	10	20	50	100
1,2-Dichlorobenzene	5.0	10	20	50	100
1,3-Dichlorobenzene	5.0	10	20	50	100
1,4-Dichlorobenzene	5.0	10	20	50	100
Dichlorobromomethane	5.0	10	20	50	100
Dichlorodifluoromethane	5.0	10	20	50	100
1,1-Dichloroethane	5.0	10	20	50	100
1,2-Dichloroethane	5.0	10	20	50	100
cis-1,2-Dichloroethene	5.0	10	20	50	100
trans-1,2-Dichloroethene	5.0	10	20	50	100
1,1-Dichloroethene	5.0	10	20	50	100
1,2-Dichloropropane	5.0	10	20	50	100
cis-1,3-Dichloropropene	5.0	10	20	50	100
trans-1,3-Dichloropropene	5.0	10	20	50	100
1,4-Dioxane	250	500	1000	2500	5000

Table A-3. Calibration Levels for 624, 5 mL Purge (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Ethylbenzene	5.0	10	20	50	100
Hexane	5.0	10	20	50	100
2-Hexanone	20	40	80	200	400
Methylene chloride	5.0	10	20	50	100
4-Methyl-2-pentanone (MIBK)	20	40	80	200	400
Methyl *tert*-butyl ether (MTBE)	5.0	10	20	50	100
Styrene	5.0	10	20	50	100
1,1,1,2-Tetrachloroethane	5.0	10	20	50	100
1,1,2,2-Tetrachloroethane	5.0	10	20	50	100
Tetrachloroethene	5.0	10	20	50	100
Toluene	5.0	10	20	50	100
1,1,1-Trichloroethane	5.0	10	20	50	100
1,1,2-Trichloroethane	5.0	10	20	50	100
Trichloroethene	5.0	10	20	50	100
Trichlorofluoromethane	5.0	10	20	50	100
1,2,3-Trichloropropane	5.0	10	20	50	100
Vinyl acetate	5.0	10	20	50	100
Vinyl chloride	5.0	10	20	50	100
m- and p-Xylenes	10	20	40	100	200
o-Xylene	5.0	10	20	50	100

If the response factor (RF) is constant over the working range (<35% RSD), the average RF may used for calculations. Alternatively, a calibration curve may be used if the correlation coefficient is ≥ 0.99.

APPENDIX A

Modifications for Analysis of 1,4-Dioxane, 1,2,3-Trichloropropane, 1,2-Dibromo-3-chloropropane, and 1,2-Dibromoethane by Selected Ion Monitoring

1.0 REQUIREMENTS FOR METHOD 8260 SELECTED ION MONITORING (SIM)

1.1 The gas chromatograph/mass spectrometer (GCMS) is utilized in the SIM mode to obtain lower reporting limits. The standard analyte list and reporting limits are listed in Table Ap-1.

1.2 This method can be applied to aqueous and solid matrices.

1.3 The sample preparation is the same as defined in section 10.1.1 through 10.1.3 in this SOP, DV-MS-0010.

1.4 The tune period for this method is defined as 12 hours. Instrument tuning is described in section 10.1.11.3 above.

1.5 Initial calibration curve requirements are as follows:

1.5.1 Same as for 8260 detailed in Section 10.1.12 of this SOP.

1.5.2 The calibrations levels are shown in Table Ap-2.

1.6 Continuing calibration verification requirements are as follows:

1.6.1 The %drift for 1,4-dioxane must be $\leq$ 25% for the continuing calibration to be valid.

1.6.2 In addition, the %drift for the surrogate compounds should be $\leq$ 25%.

1.7 Matrix Spike and LCS requirements are as follows:

1.7.1 The spike levels are listed in Table Ap-3.

1.8 Internal Standards: The internal standard concentrations are listed in Table Ap-5.

1.9 Surrogates: The surrogate concentrations are listed in Table Ap-4.

1.10 Instrument Conditions are shown in Table Ap-7.

Table Ap-1.

TAL Method 8260SIM Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L	Solid, µg/Kg
1,4-Dioxane	123-91-1	2.0	5.0
1,2-Dibromo-3-chloropropane	96-12-8	0.02	1.0
1,2-Dibromoethane	106-93-4	0.02	1.0
1,2,3-Trichloropropane	96-18-4	0.02	1.0

Table Ap-2.

Method 8260SIM Calibration Levels

Calibration Level	1,4-Dioxane Aqueous Calibration Concentration, µg/L	EDB,DBCP,TCP Aqueous Calibration Concentration, µg/L	1,4-Dioxane Solid Calibration Concentration, µg/Kg	EDB,DBCP,TCP Solid Calibration Concentration, µg/Kg
1	NA	0.02	1.0	1.0
2	NA	0.05	2.0	2.0
3	0.2	0.2	4.0	4.0
4	1.0	1.0	8.0	8.0
5	2.0	2.0	16.0	16.0
6	5.0	5.0	32.0	32.0
7	10.0	10.0	48.0	48.0
8	20.0	20.0	NA	NA
SSV	5.0	5.0	16.0	16.0

Table Ap-3.

Method 8260SIM LCS Spike Concentrations

LCS Compounds	Aqueous Spiking Level, µg/L	Solid Spiking Level, µg/Kg
1,4-Dioxane	5.0	20
1,2-Dibromo-3-chloropropane	1.0	8
1,2-Dibromoethane	1.0	8
1,2,3-Trichloropropane	1.0	8

Table Ap-4.

8260SIM Surrogate Compounds

Surrogate Compounds	Aqueous Spiking Level, µg/L[1]	Solid Spiking Level, µg/Kg[1]
Dibromofluoromethane	12.5	50
1,2-Dichloroethane-d_4	12.5	50
Toluene-d_8	12.5	50
4-Bromofluorobenzene	12.5	50

[1] – Exact spike levels are dependent upon the calibration of the autosampler loop used for the addition of the surrogate spike solution.

Table Ap-5.

8260SIM Internal Standard Compounds

Surrogate Compounds	Aqueous Spiking Level, µg/L	Solid Spiking Level, µg/Kg
Fluorobenzene	12.5	50
Chlorobenzene-d_5	12.5	50
1,4-Dichlorobenzene-d_4	12.5	50

Table Ap-6.

8260 Selected Masses

Compound	Quant	Qualifier Ion
1,4-Dioxane	88	58
Fluorobenzene	96	70
Chlorobenzene-d_5	119	117
1,4-Dichlorobenzene-d_4	152	150
Dibromofluoromethane	111	113
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70
4-Bromofluorobenzene	95	174
1,2-Dibromo-3-chloropropane	157	155
1,2-Dibromoethane	107	109
1,2,3-Trichloropropane	110	75

Table Ap-7.

Suggested Instrument Conditions for 8260SIM

Selected Masses:	See Table Ap-6
Dwell Time:	≥ 30 milliseconds
Initial Column Temperature/Hold Time:	50 °C for 2 minutes
Column Temperature Program:	50 - 160 °C at 30°C/min, 160 - 220 °C at 60°C/min .
Final Column Temperature/Hold Time:	220 °C/4.3 min hold
Injector Temperature:	220 °C
Transfer Line Temperature:	260 °C
Source Temperature:	240 °C
Trap Desorb Temperature:	270 °C
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.3mL/min.
Column:	DB-624 Capillary 60m x 0.25mm x 1.8 um film thickness, or equivalent

APPENDIX B

Modifications for Analysis of Soils Collected for the State of Alaska

1. Collection and Preservation Requirements

Preservation and Holding Time for Volatiles in Soil
Method 5035A for Alaska

Container/Contents[1]	Preservation	Holding time	Analysis
Vial containing methanol and TFT surrogate	Sample is extruded into pre-tared 4 oz jar, containing 25 mL of methanol spiked with 2.5 ppm (ug/mL) of α, α, α-trifluorotoluene, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

Sample weights are calculated in the laboratory by adding the received weight of the sample into the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

2. Sample Preparation for Medium-Level Analysis – Field Preserved, AK method

a. Fill a 40 mL VOA vial with reagent water ~ 42 mL (no head space), and remove 1000 µL of water using a volumetric pipette or syringe.

b. Add 1050 µL of methanol extract to the vial and immediately cap. Invert the vial to ensure that there is no air bubble larger than 4 mm present. If a > 4 mm air bubble is present, re-prepare the sample.

c. Load the sample in the auto sampler and proceed to analyze against the methanol calibration curve.

d. As with water samples, surrogate and internal standard solutions are added by the autosampler (see Tables 7 and 7A in the main body of this SOP). The surrogate α, α, α-trifluorotoluene is added to the samples at the time of sampling. Recoveries for this surrogate will be reported in addition to recoveries for the surrogate compounds added at the time of analysis.

e. Prepare laboratory control samples by filling a 40 mL VOA vial with reagent water, and remove 1000 µL of water using a volumetric pipette or syringe. Add reagents as needed plus sufficient methanol for a total methanol volume of 1050 µL. The recommended concentration for the LCS is the same as the Level 5 of the initial calibration curve.

f. Remove a portion of the methanol extract for each sample and store in a clean Teflon-capped vial with no headspace at ≤ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

3. Percent Moisture Correction for Soils from the State of Alaska

A percent moisture correction is required for soil samples submitted from the state of AK to adjust the extraction final volume in order to allow for the miscible solvent effects. The following formula is used to determine the corrected final volume. This calculation is performed in the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

a. $$V_t = [V_m + (M * W_s/100)]$$

Where:

V_t = final extract volume, corrected for moisture (mL)

V_m = volume methanol used for extraction (mL)

M = moisture content of the sample (%)

W_s = aliquot of sample extracted (g)

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Dichlorodifluoromethane	75-71-8	80
Chloromethane	74-87-3	40
Bromomethane	74-83-9	40
Vinyl chloride	75-01-4	40
Chloroethane	75-00-3	40
n-Butanol	71-36-3	800
Trichlorofluoromethane	75-69-4	40
Acrolein	107-02-8	200
Acetone	67-64-1	400
Trichlorotrifluoroethane	76-13-1	400
Iodomethane	74-88-4	500
Carbon disulfide	75-15-0	40
Methylene chloride	75-09-2	40
tert-Butyl alcohol	75-65-0	800
1,1-Dichloroethene	75-35-4	40
1,1-Dichloroethane	75-34-3	40
trans-1,2-Dichloroethene	156-60-5	40
Acrylonitrile	107-13-1	400
Methyl *tert*-butyl ether (MTBE)	1634-04-4	200
Hexane	110-54-3	400
cis-1,2-Dichloroethene	156-59-2	40
1,2-Dichloroethene (Total)	540-59-0	40
Tetrahydrofuran	109-99-9	80
Chloroform	67-66-3	40
1,2-Dichloroethane	107-06-2	40
Dibromomethane	74-95-3	40
2-Butanone	78-93-3	160
1,4-Dioxane	123-91-1	2,000
1,1,1-Trichloroethane	71-55-6	40
Carbon tetrachloride	56-23-5	40
Bromodichloromethane	75-27-4	40
1,2-Dichloropropane	78-87-5	40
Isopropyl Alcohol	67-63-0	1,000
Isopropyl ether	108-20-3	200

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
cis-1,3-Dichloropropene	10061-01-5	40
Trichloroethene	79-01-6	40
Dibromochloromethane	124-48-1	40
1,2-Dibromoethane	106-93-4	40
1,2,3-Trichloropropane	96-18-4	40
1,1,2-Trichloroethane	79-00-5	40
Benzene	71-43-2	16
Ethylmethacrylate	97-63-2	80
trans-1,3-Dichloropropene	10061-02-6	40
Bromoform	75-25-2	40
4-Methyl-2-pentanone	108-10-1	160
2-Hexanone	591-78-6	160
Tetrachloroethene	127-18-4	40
Toluene	108-88-3	40
1,1,2,2-Tetrachloroethane	79-34-5	40
2-Chloroethyl vinyl ether	110-75-8	80
Vinyl acetate	108-05-4	80
Chlorobenzene	108-90-7	40
Ethylbenzene	100-41-4	40
Styrene	100-42-5	40
trans-1,4-Dichloro-2-butene	110-57-6	400
m- and p-Xylenes	179601-23-1	80
o-Xylene	95-47-6	40
Total xylenes	1330-20-7	80
1,3-Dichlorobenzene	541-73-1	40
1,4-Dichlorobenzene	106-46-7	40
1,2-Dichlorobenzene	95-50-1	40
2,2-Dichloropropane	590-20-7	40
Bromochloromethane	74-97-5	40
1,1-Dichloropropene	563-58-6	40
1,3-Dichloropropane	142-28-9	40
1-Chlorohexane	544-10-5	80
1,1,1,2-Tetrachloroethane	630-20-6	40

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Isopropylbenzene	98-82-8	40
Bromobenzene	108-86-1	40
n-Propylbenzene	103-65-1	40
2-Chlorotoluene	95-49-8	40
4-Chlorotoluene	106-43-4	40
1,3,5-Trimethylbenzene	108-67-8	40
tert-Butylbenzene	98-06-6	40
1,2,4-Trimethylbenzene	95-63-6	40
sec-Butylbenzene	135-98-8	40
4-Isopropyltoluene	99-87-6	40
n-Butylbenzene	104-51-8	40
1,2-Dibromo-3-chloropropane	96-12-8	200
1,2,4-Trichlorobenzene	120-82-1	40
Naphthalene	91-20-3	40
Hexachlorobutadiene	87-68-3	40
1,2,3-Trichlorobenzene	87-61-6	40
Propionitrile	107-12-0	400
Cylcohexanone	108-94-1	1,600
Methyl methacrylate	80-62-6	80
Acetonitrile	75-05-8	400
Methacrylonitrile	126-98-7	400
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	160
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	160
2-Pentanone	107-87-9	600
cis-1,4-Dichloro-2-butene	1476-11-5	400
Cyclohexane	110-82-7	40
Methyl acetate	79-20-9	200
Methylcyclohexane	108-87-2	160
2-Chloro-1,3-butadiene	126-99-8	80
2-Methyl-2-propanol	75-65-0	800

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
tert-Butyl ethyl ether	637-92-3	80
1,2,3-Trimethylbenzene	526-73-8	40
Ethyl acetate	141-78-6	80
Ethyl ether	60-29-7	200
Isobutyl alcohol	78-83-1	800
Dichlorofluoromethane	75-43-4	120
Tetrahydrothiophene	110-01-0	40

[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 Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
1,1,1,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,1-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethene	20	40	80	200	600	2000	4000	8000
1,1-Dichloropropene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichloropropane	20	40	80	200	600	2000	4000	8000
1,2,4-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,4-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dibromo-3-chloropropane	20	40	80	200	600	2000	4000	8000
1,2-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,3,5-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,4-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,4-Dioxane	1000	2000	4000	10000	30000	100000	200000	400000
1-Chlorohexane	20	40	80	200	600	2000	4000	8000
2,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
2-Butanone (MEK)	80	160	320	800	2400	8000	16000	32000
2-Chloro-1,3-butadiene (chloroprene)	20	40	80	200	600	2000	4000	8000
2-Chlorotoluene	20	40	80	200	600	2000	4000	8000
2-Hexanone	80	160	320	800	2400	8000	16000	32000
2-Methyl-2-propanol (tert-Butyl alcohol)	400	800	1600	4000	12000	40000	80000	160000
4-Chlorotoluene	20	40	80	200	600	2000	4000	8000
4-Isopropyltoluene	20	40	80	200	600	2000	4000	8000
4-Methyl-2-pentanone	80	160	320	800	2400	8000	16000	32000
Acetone	80	160	320	800	2400	8000	16000	32000
Acetonitrile	200	400	800	2000	6000	20000	40000	80000
Acrolein	200	400	800	2000	6000	20000	40000	80000
Acrylonitrile	200	400	800	2000	6000	20000	40000	80000

Table Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
Benzene	20	40	80	200	600	2000	4000	8000
Bromobenzene	20	40	80	200	600	2000	4000	8000
Bromoform	20	40	80	200	600	2000	4000	8000
Bromomethane	20	40	80	200	600	2000	4000	8000
Carbon tetrachloride	20	40	80	200	600	2000	4000	8000
Chlorobenzene	20	40	80	200	600	2000	4000	8000
Chlorobromomethane	20	40	80	200	600	2000	4000	8000
Chlorodibromomethane	20	40	80	200	600	2000	4000	8000
Chloroethane	20	40	80	200	600	2000	4000	8000
Chloroform	20	40	80	200	600	2000	4000	8000
Chloromethane	20	40	80	200	600	2000	4000	8000
cis-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
cis-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Cyclohexanone	20	40	80	200	300	1000	2000	4000
Dibromomethane	20	40	80	200	600	2000	4000	8000
Dichlorobromomethane	20	40	80	200	600	2000	4000	8000
Dichlorodifluoromethane	20	40	80	200	600	2000	4000	8000
Ethanol	1000	2000	4000	10000	30000	100000	200000	400000
Ethylbenzene	20	40	80	200	600	2000	4000	8000
Ethylene dibromide (EDB)	20	40	80	200	600	2000	4000	8000
Hexachlorobutadiene	20	40	80	200	600	2000	4000	8000
Iodomethane	20	40	80	200	600	2000	4000	8000
Isopropyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Isopropyl ether	100	200	400	1000	3000	10000	20000	40000
Isopropylbenzene	20	40	80	200	600	2000	4000	8000
m- and p-Xylenes	40	80	160	400	1200	4000	8000	16000
Methacrylonitrile	200	400	800	2000	6000	20000	40000	80000
Methylene chloride	20	40	80	200	600	2000	4000	8000
Naphthalene	20	40	80	200	600	2000	4000	8000
n-Butanol	600	1200	2400	6000	18000	60000	120000	240000
n-Butylbenzene	20	40	80	200	600	2000	4000	8000
n-Propylbenzene	20	40	80	200	600	2000	4000	8000
o-Xylene	20	40	80	200	600	2000	4000	8000
Propionitrile	200	400	800	2000	6000	20000	40000	80000
sec-Butylbenzene	20	40	80	200	600	2000	4000	8000
Styrene	20	40	80	200	600	2000	4000	8000

Table Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
tert-Butylbenzene	20	40	80	200	600	2000	4000	8000
Tetrachloroethene	20	40	80	200	600	2000	4000	8000
Tetrahydrothiophene	20	40	80	200	600	2000	4000	8000
Toluene	20	40	80	200	600	2000	4000	8000
trans-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
trans-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Trichloroethene	20	40	80	200	600	2000	4000	8000
Trichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Vinyl chloride	20	40	80	200	600	2000	4000	8000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table Bp-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1-Trifluoro-2,2-dichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloro-1,2,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
1,2,3-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2,2-tetrafluoroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
2-Chloroethy vinyl ether	20	40	80	200	600	2000	4000	8000
2-Nitropropane	20	40	80	200	600	2000	4000	8000
2-Pentanone	80	160	320	800	2400	8000	16000	32000
3-Chloro-1-propene (Allyl chloride)	20	40	80	200	600	2000	4000	8000
Carbon disulfide	20	40	80	200	600	2000	4000	8000
cis-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Cyclohexane	20	40	80	200	600	2000	4000	8000
Dichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Ethyl acetate	40	80	160	400	1200	4000	8000	16000
Ethyl ether	20	40	80	200	600	2000	4000	8000

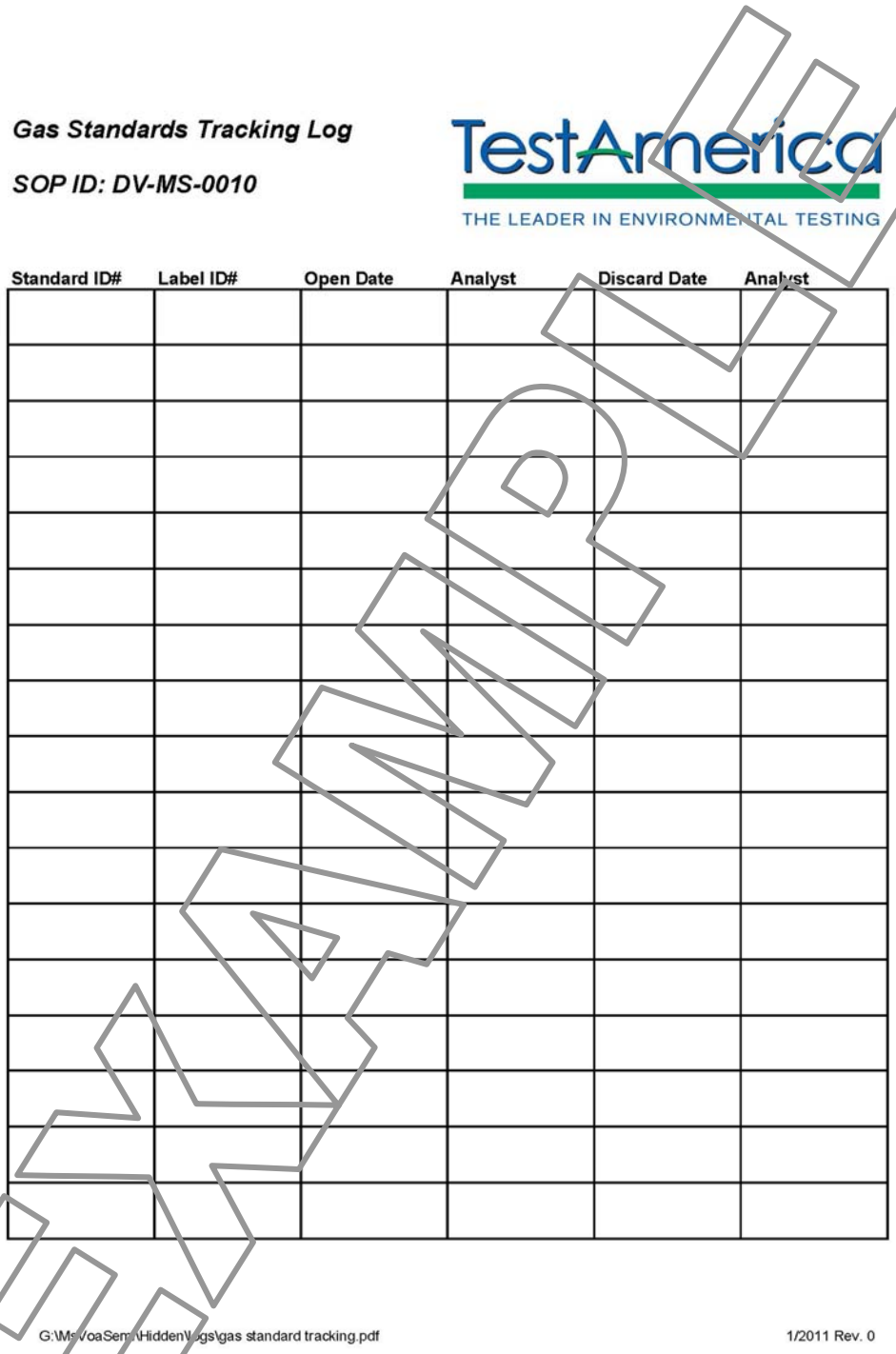
Table Bp-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Ethyl methacrylate	40	80	160	400	1200	4000	8000	16000
Ethylene oxide	2500	5000	10000	25000	75000	250000	500000	1000000
Hexane	20	40	80	200	600	2000	4000	8000
Isobutyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Methyl acetate	100	200	400	1000	3000	10000	20000	40000
Methylcylcohexane	20	40	80	200	600	2000	4000	8000
Methyl methacrylate	40	80	160	400	1200	4000	8000	16000
Methyl *tert*-butyl ether (MTBE)	20	40	80	200	600	2000	4000	8000
Propene oxide	400	800	1600	4000	12000	40000	80000	160000
sec-Butyl alcohol	600	1200	2400	6000	18000	60000	120000	240000
tert-Amyl methyl ether	100	200	400	1000	3000	10000	20000	40000
tert-Butyl ethyl ether	100	200	400	1000	3000	10000	20000	40000
Tetrahydrofuran	40	80	160	400	1200	4000	8000	16000
trans-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Vinyl acetate	40	80	160	400	1200	4000	8000	16000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Attachment 1

Gas Standards Tracking Log



Gas Standards Tracking Log

SOP ID: DV-MS-0010

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Standard ID#	Label ID#	Open Date	Analyst	Discard Date	Analyst

G:\MS\VoaSemi\Hidden\Logs\gas standard tracking.pdf 1/2011 Rev. 0

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: GC/MS Analysis Based On Methods 8270C and 625

Approvals (Signature/Date):

William Rhoades 4/26/10
William Rhoades Date
Technical Manager

Adam W. Alban 27 Apr 10
Adam Alban Date
Health & Safety Manager / Coordinator

Karen Kuoppala 4.24.10
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 4/27/10
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of wastewater following method 625.

1.1.2 The modifications presented in Appendix B may be followed for analysis of wastewater following method 8270 (best practices).

1.1.3 Direct injection of a sample may be used in limited applications.

1.1.4 Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. 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.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as ¾-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.
- Kepone should be analyzed by GC/ECD.

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.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication or Soxhlet extraction. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550
DV-OP-0010	Soxhlet Extraction of Solid Samples, SW846 3540C

2.5 Qualitative identification of the analytes 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.

3.0 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 CCCs 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 / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.4 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 QC Program document (DV-QA-003P) for further details of the batch definition.

3.5 Method Blank (MB) - 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.

3.6 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.7 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.8 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.9 Surrogates - 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, MS, and 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.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples
- Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur
- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

Details concerning cleanup steps are described in the organic extraction SOPs (see Section 2.4).

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. 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 (Section

9.3). 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.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

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.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power

before performing any maintenance.

5.1.5 The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and must be done either in a hood with the sash down to chest level or while wearing a face shield over safety glasses.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
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.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless 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 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane(Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously "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) that meets all of the criteria in Table 4 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 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.

6.7 Syringe: 10 µL or 5µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL.

6.8 Carrier gas: Ultra high-purity helium.

6.9 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Refer to tables 11 and 12 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 (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot. See Appendix B for the levels used for the 8270 best practice method.\

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). Surrogate compounds and levels are listed in Table 9.

Acid Surrogates	Base Surrogates
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-tribromophenol	Terphenyl-d4
Phenol-d5	Nitrobenzene-d5
2-chlorophenol-d4	1,2,-Dichlorobenzene-d4

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.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). LCS compounds and levels are listed in Table 7.

7.6 Matrix Spike Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in sections 7.1 to 7.6 must be refrigerated at -10°C to -20°C if it can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored at 4 ± 2 °C. The standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet QC criteria. The continuing calibration standard is stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	1 liter amber	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	4oz Jar	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	N/A

9.0 Quality Control

9.1 Initial Performance Studies

9.1.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Section 13 for a discussion of detection limit studies.

9.1.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined

every 6 months. The recovery limits are the mean recovery ±3 standard deviations for surrogates, MS, and LCS. Precision limits for the MS/MSD pair results is the absolute value of the mean relative percent difference (RPD) +3 standard deviations.

9.2.2 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is 4x or more.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS or other database so that accurate historical control limits can be generated. For multiple dilutions reported from the same extract, surrogates will be reported for all dilutions of less than 4x. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.4 Refer to the Quality Assurance Program document, DV-QA-003P, for further details of control limits.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Instrument Blank

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.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every

batch of samples. The LCS is spiked with the compounds listed in Table 8 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 µg/L, depending on the analyte, unless a special QAS states a specific level. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See Quality Assurance Program DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, 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. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.
- If re-extraction and reanalysis of the batch are 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 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (See Tables 9 and 10). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See Quality Assurance Program Policy DV-QA-003P for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.7 Surrogates

9.7.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at either 100 or 150 ug/L, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9. For the Best Practice method, see table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then 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 reveals a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

NOTE: 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.

9.7.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.7.3 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).

9.7.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.8 Nonconformance and Corrective Action

9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor 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 (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP DV-QA-0031.

9.8.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. and approved by a supervisor and QA Manager.

9.8.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.9 Quality Assurance Summaries (QAS) or Program Distillations

Certain clients may require specific project or program QC that may supersede the requirements presented in this section. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 TestAmerica Quality Assurance Program

Details of the TestAmerica Denver Quality Assurance Program, including corrective action guidelines, are presented in DV-QA-003P, Quality Assurance Program. Refer to this document if in doubt regarding corrective actions.

10.0 Procedure

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550

10.2 Sample Analysis Procedure

10.2.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.2.3 Add internal standard to an aliquot of the extract to result in a 40-ng/µL concentration (for example, 20 µL of internal standard solution at, 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.2.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.2.5 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 verification.

10.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-0033, Acceptable Manual Integration Practices) or automatically by the data system. The minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

10.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

10.2.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.2.

10.3 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, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.3.1 Guidance for Dilutions Due to Matrix

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

significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. 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.

10.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, freeze them at <-10°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.5 Retention Time Criteria for Samples

10.5.1 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.

10.5.2 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.

10.6 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 SOP DV-WC-0023 for determination of percent moisture.

10.7 Procedural Variations

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

10.8 Troubleshooting Guide

10.8.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix D, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.

- Perform mass calibration as necessary.

10.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the "top board" or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 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 the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 4.

11.3.1 Inject 25 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. 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.

11.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is < 3.0 and pentachlorophenol is < 5.0. DDT breakdown must be <20%. Refer to section 12 for the appropriate calculations.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)*.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 5. 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.

11.4.3 Compounds are assigned to the IS with the closest retention time.

11.4.4 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest when average response factors or linear

regression curve fits are used. 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.

11.4.5 For AFCEE projects, the five calibration levels will be those shown in Table 10. The table also lists a sixth calibration level that is used if a second-order regression fit is needed. The only exceptions would be for the AFCEE projects requiring special reporting limits, i.e., reporting limits different than those in the AFCEE program QAPP. Additional calibration points may be required for special projects.

11.4.6 Rejection of Calibration Points

11.4.6.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.6.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.7 Add the internal standard mixture to result in a 40-ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard). The concentrations of all analytes are listed in Tables 11 and 12. For the Best Practice method, see Table B-5 in Appendix B.

11.4.8 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response

factors, and the percent RSD of the response factors for each compound using the equations in section 12. Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.9 and 11.4.10 are met. No sample analysis may be performed unless these criteria are met.

11.4.9 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.

11.4.9.1 SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

11.4.10 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 check.

11.4.10.1 If none of the CCCs are required analytes, then project-specific calibration specifications (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.4.10.2 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

11.4.11 If the average of all RSDs in the initial calibration is < 15%, then all analytes may use average response factor for calibration.

NOTE: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

11.4.11.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary

calibration reports can be generated, then the analyst should evaluate analytes with RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

11.4.11.2 If the average of all the RSDs in the initial calibration is > 15%, then calibration using a curve fit, must be used for those analytes with RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

11.4.11.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.990, and r square (r2) greater than 0.9801 Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990.

Note: South Carolina can only be analyzed using linear calibration.

11.4.11.4 An initial calibration verification containing all components from a second source (an alternate vendor, or, a unique lot from the same vendor, or, the same source but prepared by an alternate analyst) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 75-125% for DoD projects (e.g., AFCEE); 65-135% for non-DoD projects (e.g., 625/8270C HSL components); and 45-155% for poor performers (e.g., 8270C AP9, Custom, Refinery, DBP, benzaldehyde).

11.4.12 Weighting of Calibration 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/Concentration2 weighting (often called 1/X2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Because the data system does not indicate the type of weighting used, the analyst must make a notation on the initial calibration form as to the weighting used (e.g. $1/x$ or $1/x^2$).

11.4.13 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, Section 11.5.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 4.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the CCV.

11.5.3 The following criteria must be met for the CCV to be acceptable:

- The SPCC compounds must have a response factor ≥ 0.050.
- The percent difference or drift (%D) of the CCC compounds must be ≤ 20%. (See Section 12 for calculations.)
- For compounds of interest, reliably performing compounds (see Table 14, List 1 Reliably Performing Compounds) should have a %D ≤ 35%. Poorly performing compounds (see Table 15, List 2 Poorly Performing Compounds) should have a %D ≤ 50%, with allowance for up to 6 target analytes to have %D values greater than the applicable limit. Any compound of interest that does not meet the applicable criteria will be narrated.
- The internal standard response of the CCV must be within 50 - 200% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

11.5.3.1 If none of the CCCs are required analytes, project-specific calibration requirements (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.5.4 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 or equal to 12 hours after the DFTPP is acceptable.)

NOTE: Some states (like Arizona) have special requirements. Please refer to the posted QAS.

12.0 Calculations / Data Reduction

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 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.

12.1.1 The sample component relative retention time must compare to within ± 0.06 RRT units of the relative 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 characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.4 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%.)

12.1.5 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.

12.2 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. Following are guidelines for making tentative identification:

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: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should 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 the presence of co-eluting compounds.

12.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that all isomers have distinct retention times.

12.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Ariazine
Famphur
Benzidine

12.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound

SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RFi = RF for each of the calibration levels

n = Number of RF values

12.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

Cactual = Known concentration in standard

Cfound = Measured concentration using selected quantitation method

12.7 Calculating the 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.

12.7.1 Average Response Factor Calibration

If the average of all the RSDs of the response factors in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

Cex = Concentration in the extract, µg/mL

Rx = Response for the analyte

Ris = Response for the internal standard

Cis = Concentration of the internal standard

$\overline{RF}$ = Average response factor

12.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept of linear calibration line
B	=	Slope of linear calibration line

12.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept
B	=	Factor for the linear term of the quadratic calibration function
C	=	Factor for the curvature term of the quadratic calibration function

12.8 Calculating the Concentration in the Sample

12.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

Cex	=	Concentration in the extract
Vt	=	Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)
Vo	=	Volume of the sample that was extracted (mL)

12.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

Cex = Concentration in the extract

Vt = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)

Ws = 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

12.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Area of the characteristic ion for the compound being measured

Ais = Area of the characteristic ion for the specific internal standard

Cx = Concentration of the compound being measured (µg/L)

Cis = Concentration of the specific internal standard (µg/L)

12.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (12.11) with the following exceptions:

Ax = Area of the total ion chromatogram for the compound being measured

Ais = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.13 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

12.14 Calculating the Peak Tailing Factor

$$TailingFactor = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that .

AC = AB + BC, with

AB = left-hand segment

BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows

13.1.1 Prepare seven replicates at three to five times the estimated MDL concentration.

13.1.2 Extract and analyze the MDL standards as described in Section 10.

13.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

13.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.

13.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

13.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria:	The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio ≥ 3, under routine instrument conditions.
Corrective Actions:	If the first MDLV is not detected, the MDLV standard will be reprepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

13.3.1 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 a mid-level calibration.

13.3.2 Calculate the mean recovery and standard deviation for each analyte of interest.

13.3.3 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.

13.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

13.4.1 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. Further details concerning the training program are described in SOP DV-QA-0024.

13.4.2 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 should include the analysis of an extracted standard at the reporting limit and a single point calibration.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride- B

15.2.3 Flammable Solvent- Waste Stream C

15.2.4 Used vials- Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 40CFR, part 136, Appendix A, "Base/Neutrals and Acids", Method 625.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes 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.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3 This procedure includes the option for weighted linear regression curves using 1/concentration2 weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on 1/standard deviation2 weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance (standard deviation2) is proportional to the standard concentration. EPA accepted this argument and issued a letter in July 1998, which authorizes the use of 1/concentration2 weighting factors.

18.0 Attachments

Table 1. TestAmerica Primary Standard and Standard Reporting Limits
Table 2. TestAmerica Appendix IX Standard Reporting Limits
Table 3. Suggested Instrument Conditions
Table 4. DFTPP Key Ions and Ion Abundance Criteria
Table 5. Characteristic Ions, Primary Standard (in approximate retention time order)
Table 6. Characteristic Ions, Appendix IX Standard (in approximate retention time order)
Table 7. 8270C LCS Compounds
Table 8. TCLP LCS Compounds
Table 9. 8270C Surrogate Compounds
Table 10. Calibration Levels for AFCEE Projects, µg/mL
Table 11. Calibration Levels, Primary Standard, µg/mL
Table 12. Calibration Levels, Appendix IX Standard, µg/mL
Table 13. Initial Demonstration Recovery and Precision Limits
Table 14. List 1 Reliably Performing Compounds
Table 15. List 2 Poorly Performing Compounds
APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 625
Table A-1. TestAmerica Method 625 Standard Reporting List and Reporting Limits
Table A-2. Method 625 LCS and MS Compounds and Spike Concentrations
APPENDIX B. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)
Table B-1. TestAmerica Method 8270BP Standard Reporting Limits
Table B-2. Method 8270BP Calibration Levels
Table B-3. Method 8270BP LCS Spike Concentrations
Table B-4. 8270BP Surrogate Compounds
Table B-5 8270BP Internal Standard Compounds
Table B-6. Suggested Instrument Conditions for 8270BP

APPENDIX C. Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 5.2, dated 04 May 2010
  - Annual Review
  - Added section 6.9.

- Revision 5.1, dated 17 April 2009
  - Updated Table 8 to contain a longer list of LCS compounds.
  - Corrected several references to incorrect sections.
  - Removed all references to the isotope dilution method.
  -
- Revision 5, dated 20 March 2008
  - Integration for TestAmerica and STL operations.
  - Revised Tables 1 and 2 to reflect current reporting limits.
  - Removed the use of average average from the calibration section 11.4.10.

Changes fromPrevious Major Revision

- Removed the modifications for 1,4-dioxane by isotope dilution, and included this compound in Appendix B, 8270 Best Practice.

Table 1.

TAL Primary Standard 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	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	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	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,4-Dinitrotoluene	121-14-2	10	330

Table 1.

TAL Primary Standard and Standard Reporting Limits (cont.)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
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	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	108-39-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard is the standard normally used at TAL. 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.

TAL Appendix IX 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
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate	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	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
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	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330

Table 2.

TAL Appendix IX Standard Reporting Limits (cont.)

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.

Suggested Instrument Conditions

Mass Range:	35 - 500 amu
Scan Time:	≤ 1 second/scan
Initial Column Temperature/Hold Time:	40 °C for 1 minute
Column Temperature Program:	40 - 325 °C at 25 °C/min.
Final Column Temperature/Hold Time:	325 °C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C
Transfer Line Temperature:	290 °C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, split / splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 3.4 mL/min.

Table 4.

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 - 100% of mass 198
443	17 - 23% of mass 442

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
1,4-Dioxane	88	58	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	77
2,2'-oxybis(1-chloropropane)[1]	45	77	79
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
Nitrobenzene	77	123	65
Isophorone	82	95	138
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
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
Hexachlorocyclopentadiene	237	235	271
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

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
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	109	139	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	105	51
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	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Famphur	218	93	125
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
4,4-Methylenebis(2-Chloroanaline)	231	266	-
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
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 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order)

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
3/4-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	54
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	50	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
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	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	115
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
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

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 7.

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
2-Methylnaphthalene	100
Carbazole	100
Anthracene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150
2,4,6-Trichlorophenol	150
2-Methylphenol	150

Table 8.

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA group.

Table 9.

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

1. Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10.

Calibration Levels for AFCEE Projects, µg/mL

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
Pyridine	20	50	80	120	200	---	160
N-nitrosodimethylamine	10	20	50	80	120	200	---
Aniline	10	20	50	80	120	200	---
Phenol	10	20	50	80	120	200	---
Bis(2-chloroethyl)ether	10	20	50	80	120	200	---
2-Chlorophenol	10	20	50	80	120	200	---
1,3-Dichlorobenzene	10	20	50	80	120	200	---
1,4-Dichlorobenzene	10	20	50	80	120	200	---
Benzyl alcohol	10	20	50	80	120	200	---
1,2-Dichlorobenzene	10	20	50	80	120	200	---
2-Methylphenol	10	20	50	80	120	200	---
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	200	---
4-Methylphenol	10	20	50	80	120	200	---
N-Nitroso-di-n-propylamine	10	20	50	80	120	200	---
Hexachloroethane	10	20	50	80	120	200	---
Nitrobenzene	10	20	50	80	120	200	---
Isophorone	10	20	50	80	120	200	---
2-Nitrophenol	10	20	50	80	120	200	---
2,4-Dimethylphenol	10	20	50	80	120	200	---
Benzoic acid	50	50	80	120	200	---	160
Bis(2-chloroethoxy)methane	10	20	50	80	120	200	---
2,4-Dichlorophenol	10	20	50	80	120	200	---
1,2,4-Trichlorobenzene	10	20	50	80	120	200	---
Naphthalene	10	20	50	80	120	200	---
4-Chloroaniline	10	20	50	80	120	200	---
Hexachlorobutadiene	20	20	50	80	120	200	---
4-Chloro-3-methylphenol	10	20	50	80	120	200	---
2-Methylnaphthalene	10	20	50	80	120	200	---
Hexachlorocyclopentadiene	20	50	80	120	200	---	160
2,4,6-Trichlorophenol	10	20	50	80	120	200	---
2,4,5-Trichlorophenol	10	20	50	80	120	200	---
2-Chloronaphthalene	10	20	50	80	120	200	---
2-Nitroaniline	20	50	80	120	200	---	160
Dimethyl phthalate	10	20	50	80	120	200	---
Acenaphthylene	10	20	50	80	120	200	---
3-Nitroaniline	20	50	80	120	200	---	160
Acenaphthene	10	20	50	80	120	200	---
2,4-Dinitrophenol	20	50	80	120	200	---	160
4-Nitrophenol	20	50	80	120	200	---	160
Dibenzofuran	10	20	50	80	120	200	---
2,4-Dinitrotoluene	10	20	50	80	120	200	---
2,6-Dinitrotoluene	10	20	50	80	120	200	---
Diethylphthalate	10	20	50	80	120	200	---

Table 10.

Calibration Levels for AFCEE Projects, µg/mL (cont.)

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
4-Chlorophenyl phenyl ether	10	20	50	80	120	200	---
Fluorene	10	20	50	80	200	200	---
4-Nitroaniline	20	50	80	120	200	200	---
4,6-Dinitro-2-methylphenol	20	50	80	120	200	200	---
N-Nitrosodiphenylamine	10	20	50	80	200	200	---
Azobenzene[2]	10	20	50	80	200	200	---
4-Bromophenyl phenyl ether	10	20	50	80	200	200	---
Hexachlorobenzene	10	20	50	80	200	200	---
Pentachlorophenol	20	50	80	120	200	200	---
Phenanthrene	10	20	50	80	200	200	---
Anthracene	10	20	50	80	200	200	---
Carbazole	10	20	50	80	200	200	---
Di-n-butyl phthalate	10	20	50	80	200	200	---
Fluoranthene	10	20	50	80	200	200	---
Benzidine	50	50	80	120	200	200	---
Pyrene	10	20	50	80	200	200	---
Butyl benzyl phthalate	10	20	50	80	200	200	---
3,3'-Dichlorobenzidine	10	50	80	120	200	200	---
Benzo(a)anthracene	10	20	50	80	200	200	---
Bis(2-ethylhexyl)phthalate	10	20	50	80	200	200	---
Chrysene	10	20	50	80	200	200	---
Di-n-octylphthalate	10	20	50	80	200	200	---
Benzo(b)fluoranthene	10	20	50	80	200	200	---
Benzo(k)fluoranthene	10	20	50	80	200	200	---
Benzo(a)pyrene	10	20	50	80	200	200	---
Indeno(1,2,3-cd)pyrene	10	20	50	80	200	200	---
Dibenz(a,h)anthracene	10	20	50	80	200	200	---
Diethyl phthalate	10	20	50	80	200	200	---
Benzo(g,h,i)perylene	10	20	50	80	200	200	---

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 11.

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	--	20	50	80	120	160	200
N-nitrosodimethylamine	10	20	50	80	120	160	200
Aniline	10	20	50	80	120	160	200
Phenol	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	10	20	50	80	120	160	200
2-Chlorophenol	10	20	50	80	120	160	200
1,3-Dichlorobenzene	10	20	50	80	120	160	200
1,4-Dichlorobenzene	10	20	50	80	120	160	200
Benzyl alcohol	10	20	50	80	120	160	200
1,2-Dichlorobenzene	10	20	50	80	120	160	200
2-Methylphenol	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	160	200
4-Methylphenol	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	10	20	50	80	120	160	200
Hexachloroethane	10	20	50	80	120	160	200
Nitrobenzene	10	20	50	80	120	160	200
Isophorone	10	20	50	80	120	160	200
2-Nitrophenol	10	20	50	80	120	160	200
2,4-Dimethylphenol	10	20	50	80	120	160	200
Benzoic acid	--	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	10	20	50	80	120	160	200
2,4-Dichlorophenol	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	10	20	50	80	120	160	200
Naphthalene	10	20	50	80	120	160	200
4-Chloroaniline	10	20	50	80	120	160	200
Hexachlorobutadiene	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	10	20	50	80	120	160	200
2-Methylnaphthalene	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	20	50	80	120	160	200
2,4,6-Trichlorophenol	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	10	20	50	80	120	160	200
2-Chloronaphthalene	10	20	50	80	120	160	200
2-Nitroaniline	--	20	50	80	120	160	200
Dimethyl phthalate	10	20	50	80	120	160	200
Acenaphthylene	10	20	50	80	120	160	200
3-Nitroaniline	--	20	50	80	120	160	200
Acenaphthene	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	20	50	80	120	160	200
4-Nitrophenol	--	20	50	80	120	160	200
Dibenzofuran	10	20	50	80	120	160	200
2,4-Dinitrotoluene	10	20	50	80	120	160	200
2,6-Dinitrotoluene	10	20	50	80	120	160	200
Diethylphthalate	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	10	20	50	80	120	160	200

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluorene	10	20	50	80	120	160	200
4-Nitroaniline	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	20	50	80	120	160	200
N-Nitrosodiphenylamine	10	20	50	80	120	160	200
Azobenzene[2]	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	10	20	50	80	120	160	200
Hexachlorobenzene	10	20	50	80	120	160	200
Pentachlorophenol	--	20	50	80	120	160	200
Phenanthrene	10	20	50	80	120	160	200
Anthracene	10	20	50	80	120	160	200
Carbazole	10	20	50	80	120	160	200
Di-n-butyl phthalate	10	20	50	80	120	160	200
Fluoranthene	10	20	50	80	120	160	200
Benzidine	--	20	50	80	120	160	200
Pyrene	10	20	50	80	120	160	200
Butyl benzyl phthalate	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	20	50	80	120	160	200
Benzo(a)anthracene	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline)	10	20	50	80	120	160	200
Chrysene	10	20	50	80	120	160	200
Di-n-octylphthalate	10	20	50	80	120	160	200
Benzo(b)fluoranthene	10	20	50	80	120	160	200
Benzo(k)fluoranthene	10	20	50	80	120	160	200
Benzo(a)pyrene	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	10	20	50	80	120	160	200
Diethyl phthalate	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	10	20	50	80	120	160	200

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 12.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
Acetophenone	10	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
3-Methylphenol	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
o,o,o-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
1,3-Dinitrobenzene	10	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	--	20	50	80	120	160	200
5-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 12.

Calibration Levels, Appendix IX Standard, µg/mL (cont.)

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 13.

Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 13.

Initial Demonstration Recovery and Precision Limits (cont.)

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270C.

Table 14.

List 1 Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.

List 2 Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4"-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-Dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimtheylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	o,o,o-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

APPENDIX A

Modifications Required for Analysis of Wastewater Following Method 625

REQUIREMENTS FOR METHOD 625

- Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analyte list and reporting limits are listed in Table A-1.
- This method can be applied to only aqueous matrices.
- The tune period for this method is defined as 24 hours.
- Initial calibration curve requirements are as follows:
  - The initial calibration curve for this method requires at least three points.
  - Target compounds must have RSD ≤ 35%.
  - If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.
- Continuing calibration verification requirements are as follows:
  - All target compounds must have %D ≤ 20%.
- Matrix Spike and LCS requirements are as follows:
- A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1.

TAL 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	20
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
Pentachlorophenol	87-86-5	50

Table A-1.

TAL Method 625 Standard Reporting List and Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
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
N-Nitrosodimethylamine	62-75-9	10

Table A-2.

Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
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 (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
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
N-Nitrosodimethylamine	100

APPENDIX B

Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data.. The standard analyte list and reporting limits are listed in Table B-1.
- This method can be applied to only aqueous matrices.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table B-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except the level 7 calibration point is used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table B-3.
- Internal Standards: The internal standard concentrations are listed in Table B-5.
- Surrogates: The surrogate concentrations are listed in Table B-4.
- Instrument Conditions are shown in Table B-6.

Table B-1.

TAL Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-oxybis(1-chloropropane)[2]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5
4-Bromophenyl phenyl ether	101-55-3	5

Table B-1.

TAL Method 8270BP Standard Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table B-2.

Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.
8	12.5
9	20.0
10	40.0
SSV	5.0

Table B-3.

Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table B-3.

Method 8270BP LCS Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table B-4.

8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	5
2-Fluorobiphenyl	5
Terphenyl-d14	5
1,2-Dichlorobenzene-d4[1]	5
Phenol-d5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d4[1]	7.5

Table B-5.

8270BP Internal Standard Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	5
2-Fluorobiphenyl	5
Terphenyl-d14	5
1,2-Dichlorobenzene-d4[1]	5
Phenol-d5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d4[1]	7.5

Table B-6.

Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minutes
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C/4 min hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner /splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.0mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	Weekly	As Needed	Quarterly	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 vacuum, relays, gas pressures, and flows.**	**Replace the exhaust filters on the mechanical rough pump every 1 to 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.**		**Change the oil in the mechanical rough pump.**
Check inlets, septa.		**Clean source, including all ceramics and lenses. 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.**		**Relubricate the turbomolecular pump-bearing wick.**
Check baseline level.		**Repair/replace jet separator.**		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		**Replace filaments when both filaments burn out or performance indicates the need for replacement.**		

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph (cont.)

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	*As Needed*
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 guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).***
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.***
Check inlets, septa. ***Clean injector port.***	***Replace septa.***
Check baseline level.	***Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).***
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	***Repair or replace flow controller if constant gas flow cannot be maintained.***
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Title: GC/MS Analysis Based On Methods 8270D

Approvals (Signature/Date):

Joel Kempton 12/31/12
Technical Specialist — Date

Adam Alban 1/1/13
Health & Safety Manager / Coordinator — Date

John P. Morris 1/3/13
Quality Assurance Manager — Date

William S. Cicero 1/3/13
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270D, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of samples following method 8270 (best practices).

NOTE: The 8270 Best Practice method is NOT applicable for the analysis of South Carolina regulatory compliance samples.

1.1.2 Direct injection of a sample may be used in limited applications.

1.1.3 Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. 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.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3/4-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.
- Kepone should be analyzed by GC/ECD.
- Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, 3550C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550C

2.5 Qualitative identification of the analytes 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.

3.0 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. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.2 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 TAL QC Program document (DV-QA-003P) for further details of the batch definition.

3.3 Method Blank (MB) - 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.

3.4 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.5 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.6 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.7 Surrogates - 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, MS, and 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.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography (GPC) - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples (TestAmerica Denver does not have a GPC unit).

- Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur

- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

Details concerning cleanup steps are described in the organic extraction SOP DV-OP-0007.

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. 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 (Section 9.3). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If 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.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

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.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power before performing any maintenance.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless 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 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane(Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously “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) that meets all of the criteria in Table 4 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 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.

6.7 Syringe: 10 µL or 5µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL.

6.8 Carrier gas: Ultra high-purity helium.

6.9 Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\|Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Refer to tables 11 and 12 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 (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot. See Appendix B for the levels used for the 8270 best practice method.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). Surrogate compounds and levels are listed in Table 9.

Acid Surrogates	Base Surrogates
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-tribromophenol	Terphenyl-d_4
Phenol-d_5	Nitrobenzene-d_5
2-chlorophenol-d_4	1,2,-Dichlorobenzene-d_4

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.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). LCS compounds and levels are listed in Table 7.

7.6 Matrix Spike Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards are stored away from any light source at 6 °C (-10 °C recommended). The

standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet QC criteria.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	1 liter amber	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3 and SW846 Chapter 4
Soils	4oz Jar	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	SW846 Chapter 4

9.0 Quality Control

9.1 Initial Performance Studies

9.1.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Section 13 for a discussion of detection limit studies.

9.1.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined every 6 months. The recovery limits are the mean recovery ±3 standard deviations for surrogates, MS, and LCS. Precision limits for the MS/MSD pair results is the absolute value of the mean relative percent difference (RPD) +3 standard deviations.

9.2.2 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is 4x or more.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS or other database so that accurate historical control limits can be generated. For multiple dilutions reported from the same extract, surrogates will be reported for all dilutions of less than 4x. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.4 Refer to the QC program document, DV-QA-003P, for further details of control limits.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Instrument Blank

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.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with the compounds listed in Tables 7 and 8 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 80 or 120 µg/L, depending on the analyte, unless a special QAS states a specific level. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See QC Policy DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, 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. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the

problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the failed LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (See Tables 7 and 8). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See QC Policy DV-QA-003P for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed unless project specific requirements indicate alternative corrective actions. The reasons for accepting the batch must be documented. The sample results must be flagged and the nonconformance described in the case narrative.

NOTE: South Carolina requires reanalysis to confirm matrix interference.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.7 Surrogates

9.7.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at either 100 or 150 ug/L, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9. For the Best Practice method, see table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then 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 reveals a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

NOTE: The decision to reanalyze or flag the data for failed QC 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.

9.7.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

NOTE: In these circumstances, South Carolina requires re-extraction and reanalysis of the sample and MS/MSD to confirm matrix interference.

9.7.3 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. (If the re-analysis was outside holding times, both sets of results may be reported, with appropriate flags and discussion in the case narrative. Consult client and/or program specifications for reporting requirements.)

9.7.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.8 Nonconformance and Corrective Action

9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry of the sample, sample size, or other parameters. Any variation in procedure shall be completely documented

using a Nonconformance Memo (NCM). The NCM is a then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP DV-QA-0031.

9.8.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents and approved by a supervisor and QA Manager. Unless the client requests more stringent criteria than the requirements in this SOP, the deviations must be clearly documented in the report narrative and the samples flagged accordingly.

9.8.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.9 Quality Assurance Summaries (QAS) or Program Distillations

Certain clients may require specific project or program QC that may supersede the requirements presented in this section. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 TestAmerica QC Program

Details of the Denver Quality Control Program, including corrective action guidelines, are presented in SOP DV-QA-003P, *Quality Control Program*. Refer to this document if in doubt regarding corrective actions.

10.0 Procedure

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, 3550C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550C

10.2 Sample Analysis Procedure

10.2.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.2.3 Add internal standard to an aliquot of the extract to result in a 40-ng/µL concentration (for example, 20 µL of internal standard solution at, 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.2.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.2.5 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 verification.

10.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-0033, Acceptable Manual Integration Practices) or automatically by the data system. The minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

10.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

10.2.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.2.

10.2.9 The internal standard response in the sample must be within 50 - 200% of the response in the CCV.

10.2.10 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights.

10.3 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, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.3.1 Guidance for Dilutions Due to Matrix

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 significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. 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.

10.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at ≤ 6°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.5 Retention Time Criteria for Samples

10.5.1 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.

10.5.2 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.

10.6 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 SOP DV-WC-0023 for determination of percent moisture.

10.7 Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry of the sample, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.8 Troubleshooting Guide

10.8.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix B, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “top board” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 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 the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine), see Table 4. The mass spectrum is acquired with three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designated only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not co-elute with DFTPP.

11.3.1 Inject 25 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. 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.

11.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is < 2.0 and pentachlorophenol is < 2.0. DDT breakdown must be <20%. Refer to section 12 for the appropriate calculations.

11.3.3 Degradation of DDE and DDD must not exceed 20%.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, Calibration Curves *(General)*.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 5. 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.

11.4.3 Compounds are typically assigned to the IS with the closest retention time. The laboratory tries to maintain consistent internal standard references across instruments. As a result, there may be a few cases where compounds are very close to two different internal standards that this is not true.

11.4.4 Evaluation of retention times – The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

11.4.5 Prepare calibration standards at a minimum of five concentration levels for

each parameter of interest when average response factors or linear regression curve fits are used. 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.

11.4.6 For AFCEE projects, the six calibration levels will be those shown in Table 10. The table also lists a seventh calibration level that is used if a second-order regression fit is needed. The only exceptions would be for the AFCEE projects requiring special reporting limits, i.e., reporting limits different than those in the AFCEE program QAPP. Additional calibration points may be required for special projects.

11.4.7 Rejection of Calibration Points

11.4.7.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.7.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.8 Add the internal standard mixture to result in a 40-ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard). The concentrations of all analytes are listed in Tables 11 and 12. For the Best Practice method, see Table A-1 in Appendix A.

11.4.9 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound

using the equations in section 12. No sample analysis may be performed unless the following criteria are met.

11.4.10 The RSD must be < 20% for each compound of interest.

11.4.10.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with RSD > 20% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

11.4.10.2 If the RSD in the initial calibration is > 20%, then calibration using a curve fit must be used for those analytes with RSD > 20%. Linear or quadratic curve fits may be used. Use of a weighted regression is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

11.4.10.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.99. Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990. When linear regression is used, the first point of the calibration is recalculated under the new calibration, with the values agreeing within 30% of the true values.

Note: South Carolina can only be analyzed using linear calibration.

11.4.10.4 An initial calibration verification containing all components from a second source (an alternate vendor, or, a unique lot from the same vendor) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 75-125% for DoD projects (e.g., AFCEE) and 70-130% for non-DoD projects (e.g., 8270C HSL components).

Note: Several states (Arizona) and/or federal programs have special requirements. Be sure to review state QAS summaries and SOP DV-QA-024P for special requirements.

11.4.11 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or column, then repeat the calibration procedure.

11.4.12 The minimum response factor for the most common target analytes from Table 16 must be met.

11.4.13 Weighting of Calibration 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. Because the data system does not indicate the type of weighting used, the analyst must make a notation on the initial calibration form as to the weighting used (e.g. 1/x or 1/x^2).

11.4.14 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration. For additional information on calibrations see SOP CA-Q-S-005.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 4.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the CCV.

11.5.3 The following criteria must be met for the CCV to be acceptable:

- The percent difference or drift (%D) of each compound must be ≤ 20%. (See Section 12 for calculations.)

NOTE: Some states (Wisconsin) have special continuing calibration requirements when initial calibration is performed using a quadratic curve. Please refer to state specific QAS.

- Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met for more than 20% of the compounds included in the calibration, then corrective action must take place prior to the analysis for samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.
- The internal standard response of the CCV must be within 50 - 200% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial

calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

11.5.4 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 or equal to 12 hours after the DFTPP is acceptable.)

11.5.5 Each of the most common target analytes in the CCV must meet the minimum response factors listed in table 16. If they are not met, the system is evaluated, and corrective action takes place before sample analysis begins. Possible problems include 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.

12.0 Calculations / Data Reduction

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 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.

12.1.1 The sample component relative retention time must compare to within ± 0.06 RRT units of the relative 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 characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.4 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%.)

12.1.5 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.

12.2 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. Following are guidelines for making tentative identification:

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 ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 20%and 80%.)

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 the presence of co-eluting compounds.

12.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that all isomers have distinct retention times.

12.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Atrazine
Famphur
Benzidine

12.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound

SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RF_i = RF for each of the calibration levels

n = Number of RF values

12.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.7 Calculating the 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.

12.7.1 Average Response Factor Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is ≤20%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
$\overline{RF}$	=	Average response factor

12.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
A	=	Intercept of linear calibration line
B	=	Slope of linear calibration line

12.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
A	=	Intercept
B	=	Factor for the linear term of the quadratic calibration function
C	=	Factor for the curvature term of the quadratic calibration function

12.8 Calculating the Concentration in the Sample

12.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where:

C_{ex} = Concentration in the extract

V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)

V_o = Volume of the sample that was extracted (mL)

12.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

C_{ex} = Concentration in the extract

V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)

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

12.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD	=	Relative percent difference
MS_R	=	Matrix spike result
MSD_R	=	Matrix spike duplicate result

12.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

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)

12.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (12.11) 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

12.13 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

12.14 Calculating the Peak Tailing Factor

$$TailingFactor = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that:

AC = AB + BC, with

AB = left-hand segment

BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows

13.1.1 Prepare seven replicates at three to five times the estimated MDL concentration.

13.1.2 Extract and analyze the MDL standards as described in Section 10.

13.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

13.1.4 MDL studies are repeated annually, or verified quarterly, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.

13.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

13.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria:	The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio ≥ 3, under routine instrument conditions.
Corrective Actions:	If the first MDLV is not detected, the MDLV standard will be reprepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

13.3.1 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 a mid-level calibration.

13.3.2 Calculate the mean recovery and standard deviation for each analyte of interest.

13.3.3 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.

13.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

13.4.1 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. Further details concerning the training program are described in SOP DV-QA-0024.

13.4.2 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 should include the analysis of an extracted standard at the reporting limit and a single point calibration.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 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 procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Corporate Safety Manual, and HS-001, “Waste Management Program.”

15.2 The following waste streams are produced when this method is carried out

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride- B

15.2.3 Flammable Solvent- Waste Stream C

15.2.4 Used vials- Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 4, February 2007, Method 8270D.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes 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.

Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within ± 0.06 RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within ± 0.2 minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within ± 0.2 minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2 minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3 This procedure includes the option for weighted linear regression curves using $1/concentration^2$ weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on $1/standard\ deviation^2$ weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance ($standard\ deviation^2$) is proportional to the standard concentration. EPA accepted this argument and issued a memorandum dated August 7, 1998 (Attachments dated July 1998), which authorizes the use of $1/concentration^2$ weighting factors.

18.0 Attachments

Table 1. TAL Primary Standard and Standard Reporting Limits

Table 2. TAL Appendix IX Standard Reporting Limits

Table 3. Suggested Instrument Conditions

Table 4. DFTPP Key Ions and Ion Abundance Criteria

Table 5. Characteristic Ions, Primary Standard (in approximate retention time order)

Table 6. Characteristic Ions, Appendix IX Standard (in approximate retention time order)

Table 7. 8270D LCS Compounds

Table 8. TCLP LCS Compounds

Table 9. 8270D Surrogate Compounds

Table 10. Calibration Levels for AFCEE Projects, µg/mL

Table 11. Calibration Levels, Primary Standard, µg/mL

Table 12. Calibration Levels, Appendix IX Standard, µg/mL

Table 13. Initial Demonstration Recovery and Precision Limits

Table 14. List 1 Reliably Performing Compounds

Table 15. List 2 Poorly Performing Compounds

Table 16 Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

- Table A-1. TAL Method 8270BP Standard Reporting Limits
- Table A-2. Method 8270BP Calibration Levels
- Table A-3. Method 8270BP LCS Spike Concentrations
- Table A-4. 8270BP Surrogate Compounds
- Table A-5. Suggested Instrument Conditions for 8270BP

APPENDIX B. Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 3, dated 4 January 2013
  - Changed storage of extracts from freezer to refrigerator.
- Revision 2, dated 30 November 2012
  - Deleted Section 5.1.5 – About the use of Separatory Funnels
  - Updated the Hazardous Materials table in Section 5.2 to reflect current solvent used.
  - Updated and clarified language in Attachments to reflect current practices.

- Revision 1.1, dated 01 December 2011
  - Added note to section 1.1.1 and Appendix A restricting use of Best Practice method
  - Revised sections 9.5, 9.6 and 9.7 to clarify use of flags and documentation in narrative for failed QC
  - Revised section 9.8.1 to clarify modifications might be made to accommodate the chemistry of the sample.
  - Added statement to 11.5.3 to flag data if target analyte reported for failed CCV.
  - Expanded the discussion in section 17.1.1 to clarify how the use of RT windows stipulated in this SOP meets or exceeds the requirements of Method 8270D.
  - Clarified reference from EPA for source of inverse weighted least squares regression for calibrations.

- Revision 1.0, dated 31 January 2011
  - Updated Table 3, Suggested Instrument Conditions
  - Added components and changed spike levels in Table 7, 8270D LCS Compounds
  - Added low level calibration standard to Table 11, Calibration Levels, Primary Standard
  - Minor grammatical, spelling and formatting changes were made throughout.

- Revision 0.1, dated 11 December 2009
  - Added requirements for degradation criteria for DDD and DDE in the tune to section 11.3.3.
  - Removed statement allowing an ICV standard from the same vendor and lot as long as it is prepared by a separate analyst from section 11.4.10.4.

Table 1.

TAL Primary Standard 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	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	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	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,4-Dinitrotoluene	121-14-2	10	330

Table 1.

TAL Primary Standard and Standard Reporting Limits (cont.)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
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	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	108-39-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard is the standard normally used at TAL. 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 formerly known as bis(2-chloroisopropyl)ether.

Table 2.

TAL Appendix IX 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
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
O,O,O-Triethyl-Phosphorothioate	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	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
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	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600

Table 2.

TAL Appendix IX Standard Reporting Limits (cont.)

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.

Suggested Instrument Conditions[1]

Mass Range:	35 - 500 amu
Scan Time:	≤ 1 second/scan
Initial Column Temperature/Hold Time:	55 °C for 1.5 minutes
Column Temperature Program:	25 ˚C/min. to 250 °C then 5 °C/min. to 330 ˚C
Final Column Temperature/Hold Time:	330 ˚C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C
Transfer Line Temperature:	300 °C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, split / splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 3.4 mL/min.

[1]The GC parameters should be optimized to provide appropriate resolution for benzo(b)fluoranthene and benzo(k)fluoranthene and dibenz(a,h)anthracene and indeno(1,2,3-cd)pyrene.

Table 4.

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 - 100% of mass 198
443	17 - 23% of mass 442

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
N-Nitrosodimethylamine	74	42	--
1,4-Dioxane	88	58	--
Pyridine	79	52	--
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d_5 (Surrogate Standard)	99	42	71
Aniline	93	66	--
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d_4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	77
2,2'-Oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d_5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
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-d_8 (Internal Standard)	136	68	54
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
Hexachlorocyclopentadiene	237	235	271
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

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d_{10} (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	109	139	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	105	51
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-d_{10} (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d_{14} (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Famphur	218	93	125
Benzo(a)Anthracene	228	229	226
Chrysene-d_{12} (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
4,4-Methylenebis(2-Chloroanaline)	231	266	--
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d_{12} (Internal Standard)	264	260	265
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 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order)

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
3/4-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	54
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	50	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	--
2-Naphthylamine	143	115	--
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	75	97	121
Phenacetin	108	179	109
Diallate	86	234	--
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	115
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
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

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	--
Aramite 1	185	319	--
Aramite 2	185	319	--
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	--
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	--
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 7.

8270D LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
Azobenzene	80
Acetophenone	80
Acenaphthylene	80
Benzo[a]anthracene	80
Benzo[b]fluoranthene	80
Benzo[k]fluoranthene	80
Benzoic acid	80
Benzo[g,h,i]perylene	80
Benzo[a]pyrene	80
Benzyl alcohol	80
Bis(2-chloroethoxy)methane	80
Bis(2-ethylhexyl) phthalate	80
Butyl benzyl phthalate	80
Bis(2-chloroethyl)ether	80
Carbazole	80
Chrysene	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Di-n-butyl phthalate	80
Di-n-octyl phthalate	80
Dibenz(a,h)anthracene	80
Dibenzofuran	80
Diethyl phthalate	80
Dimethyl phthalate	80
Diphenylamine	80
Ethyl methanesulfonate	80
Fluoranthene	80
Fluorene	80
Hexachlorobenzene	80
Hexachlorobutadiene	80
Hexachlorocyclopentadiene	80
Hexachloroethane	80
Indeno(1,2,3-cd)pyrene	80
Isosafrole	80
Isophorone	80
Methyl methanesulfonate	80
N-Nitrodimethylamine	80
N-Nitrosodi-n-butylamine	80
N-Nitrosodiethylamine	80
N-Nitrosodi-n-propylamine	80
N-Nitrosodiphenylamine	80
N-Nitrosomethylethylamine	80
N-Nitrosomorpholine	80
N-Nitrosopiperidine	80
N-Nitrosopyrrolidine	80
Pentachlorobenzene	80
Pentachloroethane	80
Pentachloronitrobenzene	80
Pentachlorophenol	80
Phenacetin	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Phenanthrene	80
Phenol	80
Pyrene	80
Pyridine	80
Safrole, Total	80
1,4-Dichlorobenzene	80
Naphthalene	80
2-Methylnaphthalene	80
3-Methylcholanthrene	80
1-Naphthylamine	80
Nitrobenzene	80
2-Picoline	80
7,12-Dimethylbenz(a)anthracene	80
2-Fluorobiphenyl	80
2-Fluorophenol	80
2,4,6-Tribromophenol	80
Nitrobenzene-d5	80
Phenol-d5	80
Terphenyl-d14	80
1,4-Dichlorobenzene-d4	80
Acenaphthene-d10	80
Chrysene-d12	80
Naphthalene-d8	80
Phenanthrene-d10	80
Perylene-d12	80
1-Chloronaphthalene	80
2,3,4,6-Tetrachlorophenol	80
alpha,alpha-Dimethyl phenethylamine	80
Benzidine	80
Dibenz[a,j]acridine	80
p-Dimethylamino azobenzene	80
Pronamide	80
1,3,5-Trinitrobenzene	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
1,3-Dinitrobenzene	80
1,4-Dinitrobenzene	80
1,4-Naphthoquinone	80
2-Acetylaminofluorene	80
3,3'-Dimethylbenzidine	80
4-Nitroquinoline-1-oxide	80
N-Nitro-o-toluidine	80
Aramite, Total	80
Aramite Peak 1	80
Aramite Peak 2	80
1,1'-Biphenyl	80
Chlorobenzilate	80
Diallate	80
Dimethoate	80
Disulfoton	80
Hexachloropropene	80
Isodrin	80
Methapyrilene	80
Methyl parathion	80
O,O',O''-Triethylphosphorothioate	80
Ethyl Parathion	80
Phorate	80
p-Phenylenediamine	80
Sulfotepp	80
Thionazin	80
N-Nitrosodimethylamine	80
Dinoseb	80

Table 8.

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA group.

Table 9.

8270D Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d_5	100
2-Fluorobiphenyl	100
Terphenyl-d_{14}	100
1,2-Dichlorobenzene-d_4[1]	100
Phenol-d_5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d_4[1]	150

1. Included in standard mix, but not routinely evaluated for method 8270D. Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10.

Calibration Levels for AFCEE Projects, µg/mL

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
Pyridine	20	50	80	120	200	---	160
N-Nitrosodimethylamine	10	20	50	80	120	200	---
Aniline	10	20	50	80	120	200	---
Phenol	10	20	50	80	120	200	---
Bis(2-chloroethyl)ether	10	20	50	80	120	200	---
2-Chlorophenol	10	20	50	80	120	200	---
1,3-Dichlorobenzene	10	20	50	80	120	200	---
1,4-Dichlorobenzene	10	20	50	80	120	200	---
Benzyl alcohol	10	20	50	80	120	200	---
1,2-Dichlorobenzene	10	20	50	80	120	200	---
2-Methylphenol	10	20	50	80	120	200	---
2,2'-Oxybis(1-chloropropane)[1]	10	20	50	80	120	200	---
4-Methylphenol	10	20	50	80	120	200	---
N-Nitroso-di-n-propylamine	10	20	50	80	120	200	---
Hexachloroethane	10	20	50	80	120	200	---
Nitrobenzene	10	20	50	80	120	200	---
Isophorone	10	20	50	80	120	200	---
2-Nitrophenol	10	20	50	80	120	200	---
2,4-Dimethylphenol	10	20	50	80	120	200	---
Benzoic acid	20	50	80	120	200	---	160
Bis(2-chloroethoxy)methane	10	20	50	80	120	200	---
2,4-Dichlorophenol	10	20	50	80	120	200	---
1,2,4-Trichlorobenzene	10	20	50	80	120	200	---
Naphthalene	10	20	50	80	120	200	---
4-Chloroaniline	10	20	50	80	120	200	---
Hexachlorobutadiene	10	20	50	80	120	200	---
4-Chloro-3-methylphenol	10	20	50	80	120	200	---
2-Methylnaphthalene	10	20	50	80	120	200	---
Hexachlorocyclopentadiene	20	50	80	120	200	---	160
2,4,6-Trichlorophenol	10	20	50	80	120	200	---
2,4,5-Trichlorophenol	10	20	50	80	120	200	---
2-Chloronaphthalene	10	20	50	80	120	200	---
2-Nitroaniline	20	50	80	120	200	---	160
Dimethyl phthalate	10	20	50	80	120	200	---
Acenaphthylene	10	20	50	80	120	200	---
3-Nitroaniline	20	50	80	120	200	---	160
Acenaphthene	10	20	50	80	120	200	---
2,4-Dinitrophenol	20	50	80	120	200	---	160
4-Nitrophenol	20	50	80	120	200	---	160
Dibenzofuran	10	20	50	80	120	200	---
2,4-Dinitrotoluene	10	20	50	80	120	200	---
2,6-Dinitrotoluene	10	20	50	80	120	200	---
Diethylphthalate	10	20	50	80	120	200	---

Table 10.

Calibration Levels for AFCEE Projects, µg/mL (cont.)

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
4-Chlorophenyl phenyl ether	10	20	50	80	120	200	---
Fluorene	10	20	50	80	200	---	---
4-Nitroaniline	20	50	80	120	200	---	---
4,6-Dinitro-2-methylphenol	20	50	80	120	200	---	---
N-Nitrosodiphenylamine	10	20	50	80	200	---	---
Azobenzene[2]	10	20	50	80	200	---	---
4-Bromophenyl phenyl ether	10	20	50	80	200	---	---
Hexachlorobenzene	10	20	50	80	200	---	---
Pentachlorophenol	20	50	80	120	200	---	---
Phenanthrene	10	20	50	80	200	---	---
Anthracene	10	20	50	80	200	---	---
Carbazole	10	20	50	80	200	---	---
Di-n-butyl phthalate	10	20	50	80	200	---	---
Fluoranthene	10	20	50	80	200	---	---
Benzidine	20	50	80	120	200	---	---
Pyrene	10	20	50	80	200	---	---
Butyl benzyl phthalate	10	20	50	80	200	---	---
3,3'-Dichlorobenzidine	10	50	80	120	200	---	---
Benzo(a)anthracene	10	20	50	80	200	---	---
Bis(2-ethylhexyl)phthalate	10	20	50	80	200	---	---
Chrysene	10	20	50	80	200	---	---
Di-n-octylphthalate	10	20	50	80	200	---	---
Benzo(b)fluoranthene	10	20	50	80	200	---	---
Benzo(k)fluoranthene	10	20	50	80	200	---	---
Benzo(a)pyrene	10	20	50	80	200	---	---
Indeno(1,2,3-cd)pyrene	10	20	50	80	200	---	---
Dibenz(a,h)anthracene	10	20	50	80	200	---	---
Diethyl phthalate	10	20	50	80	200	---	---
Benzo(g,h,i)perylene	10	20	50	80	200	---	---

1. 2,2'-Oxybis(1-chloropropane) was formerly 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 11.

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Pyridine	--	10	20	50	80	120	160	200
N-Nitrosodimethylamine	4	10	20	50	80	120	160	200
Aniline	4	10	20	50	80	120	160	200
Phenol	4	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	4	10	20	50	80	120	160	200
2-Chlorophenol	4	10	20	50	80	120	160	200
1,3-Dichlorobenzene	4	10	20	50	80	120	160	200
1,4-Dichlorobenzene	4	10	20	50	80	120	160	200
Benzyl alcohol	4	10	20	50	80	120	160	200
1,2-Dichlorobenzene	4	10	20	50	80	120	160	200
2-Methylphenol	4	10	20	50	80	120	160	200
2,2'-Oxybis(1-chloropropane)[1]	4	10	20	50	80	120	160	200
4-Methylphenol	4	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	4	10	20	50	80	120	160	200
Hexachloroethane	4	10	20	50	80	120	160	200
Nitrobenzene	4	10	20	50	80	120	160	200
Isophorone	4	10	20	50	80	120	160	200
2-Nitrophenol	4	10	20	50	80	120	160	200
2,4-Dimethylphenol	4	10	20	50	80	120	160	200
Benzoic acid	--	10	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	4	10	20	50	80	120	160	200
2,4-Dichlorophenol	4	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	4	10	20	50	80	120	160	200
Naphthalene	4	10	20	50	80	120	160	200
4-Chloroaniline	4	10	20	50	80	120	160	200
Hexachlorobutadiene	4	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	4	10	20	50	80	120	160	200
2-Methylnaphthalene	4	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	10	20	50	80	120	160	200
2,4,6-Trichlorophenol	4	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	4	10	20	50	80	120	160	200
2-Chloronaphthalene	4	10	20	50	80	120	160	200
2-Nitroaniline	4	10	20	50	80	120	160	200
Dimethyl phthalate	4	10	20	50	80	120	160	200
Acenaphthylene	4	10	20	50	80	120	160	200
3-Nitroaniline	4	10	20	50	80	120	160	200
Acenaphthene	4	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	10	20	50	80	120	160	200
4-Nitrophenol	--	10	20	50	80	120	160	200
Dibenzofuran	4	10	20	50	80	120	160	200
2,4-Dinitrotoluene	4	10	20	50	80	120	160	200
2,6-Dinitrotoluene	4	10	20	50	80	120	160	200
Diethylphthalate	4	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	4	10	20	50	80	120	160	200

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Fluorene	4	10	20	50	80	120	160	200
4-Nitroaniline	--	10	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	10	20	50	80	120	160	200
N-Nitrosodiphenylamine	4	10	20	50	80	120	160	200
Azobenzene[2]	4	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	4	10	20	50	80	120	160	200
Hexachlorobenzene	4	10	20	50	80	120	160	200
Pentachlorophenol	--	10	20	50	80	120	160	200
Phenanthrene	4	10	20	50	80	120	160	200
Anthracene	4	10	20	50	80	120	160	200
Carbazole	4	10	20	50	80	120	160	200
Di-n-butyl phthalate	4	10	20	50	80	120	160	200
Fluoranthene	4	10	20	50	80	120	160	200
Benzidine	--	10	20	50	80	120	160	200
Pyrene	4	10	20	50	80	120	160	200
Butyl benzyl phthalate	4	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	10	20	50	80	120	160	200
Benzo(a)anthracene	4	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	4	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline)	4	10	20	50	80	120	160	200
Chrysene	4	10	20	50	80	120	160	200
Di-n-octylphthalate	4	10	20	50	80	120	160	200
Benzo(b)fluoranthene	4	10	20	50	80	120	160	200
Benzo(k)fluoranthene	4	10	20	50	80	120	160	200
Benzo(a)pyrene	4	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	4	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	4	10	20	50	80	120	160	200
Diethyl phthalate	4	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	4	10	20	50	80	120	160	200

1. 2,2'-Oxybis(1-chloropropane) was formerly 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 12.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
Acetophenone	10	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
3-Methylphenol	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
O,O,O-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
1,3-Dinitrobenzene	10	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	--	20	50	80	120	160	200
5-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 12.

Calibration Levels, Appendix IX Standard, µg/mL (cont.)

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 13.

Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(g,h,i)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
β-BHC[1]	60	31.5	41.5-130.6
δ-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 13.

Initial Demonstration Recovery and Precision Limits (cont.)

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270D, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270D.

Table 14.

List 1 Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.

List 2 Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4'-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimethylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	*O,O,O*-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

Table 16
Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

Analyte	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis(1-chloropropane)[1]	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethylphthalate	0.010
Acenaphthylene	0.900
2,6-Dinitrotoluene	0.200
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
Dibenzofuran	0.800
4-Nitrophenol	0.010
2,4-Dinitrotoluene	0.200
Diethylphthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
Fluorene	0.900
4-Chlorophenylphenylether	0.400
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
N-Nitrosodiphenylamine	0.010
4-Bromophenylphenylether	0.100

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

Table 16
Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (cont.)

Analyte	Minimum Response Factor (RF)
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
Benzo(a)anthracene	0.800
3,3'-Dichlorobenzidine	0.010
Chrysene	0.700
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octylphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

APPENDIX A

Modifications Required for Analysis of Samples Following Method 8270 Best Practice (8270BP)

NOTE: The 8270 Best Practice method is NOT applicable for the analysis of South Carolina regulatory compliance samples.

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data. The standard analyte list and reporting limits are listed in Table A-1.
- This method is only applicable to the analysis of low level samples. The appropriate range for aqueous samples is 1 to 100 ug/L, and 30 to 1650 ug/Kg for soils. Attempts to analyze samples with concentrations much higher than this for target compounds, or high concentrations of non-target compounds will likely result in a decline in the quality control parameters for the method. Once the instrument has been adversely impacted by high level samples, it may not be possible to bring it back into control in a reasonable time frame.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table A-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except that 7 calibration point levels are used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table A-3.
- Surrogates: The surrogate concentrations are listed in Table A-4.
- Instrument Conditions are shown in Table A-5.

Table A-1.

TAL Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-Nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-Oxybis(1-chloropropane)[1]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

Table A-1.

TAL Method 8270BP Standard Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
4-Bromophenyl phenyl ether	101-55-3	5
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table A-2.

Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.0
8	12.5
9	20.0
10	40.0
SSV	5.0

Table A-3.

Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-Oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table A-3.

Method 8270BP LCS Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table A-4.

8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d_5	5
2-Fluorobiphenyl	5
Terphenyl-d_{14}	5
1,2-Dichlorobenzene-d_4	5
Phenol-d_5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d_4	7.5

Table A-5.

Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minutes
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C/4 min hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner /splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.0mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX B

Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily (when used)	**Weekly**	**As Needed**	**Quarterly**	**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 vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 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.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. 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.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX B

Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph (cont.)

GAS CHROMATOGRAPH Instrument Maintenance Schedule (*For GC/MS only.*)	
Daily (when used)	***As Needed***
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 guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
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.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Title: Chemical Oxygen Demand by Method 410.4

[EPA 410.4]

Approvals (Signature/Date):

Dave Elkin — Date: 10/4/10 Wet Chemistry Supervisor	Adam Alban — Date: 04Oct10 Health & Safety Manager / Coordinator
Karen Kuoppala — Date: 10/6/10 Quality Assurance Manager	Robert C. Hanisch — Date: 10/7/10 Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

- This standard operating procedure (SOP) provides instructions for the determination of chemical oxygen demand (COD) in aqueous samples. The test determines the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant.
- This method is applicable to surface waters, and domestic and industrial wastes.
- This method covers a practical COD concentration range of 10 - 200 mg/L. Higher concentrations can be determined by dilution of the sample. The reporting limit is 10 mg/L.

2.0 Summary of Method

2.1 This method is based on reduction of Cr+6 to Cr+3. The COD tubes contain a pre-measured concentration of Cr+6. COD in the samples causes Cr+6 to be reduced, so that the Cr+6 concentration decreases with increasing COD concentration. The absorbance at 420 nm is proportional to the Cr+6 concentration and inversely proportional to COD concentration.

2.2 COD is related to TOC and BOD in typical samples consisting of readily oxidized materials. COD should be higher than BOD, and BOD should be higher than TOC. The exact ratios depend on the actual composition of the samples. For EPA and similar QC samples, the ratios are approximately BOD = 0.6 x COD and TOC = 0.4 X COD.

3.0 Definitions

3.1 **COD:** "Chemical Oxygen Demand" is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions.

3.2 **TOC:** "Total Organic Carbon" is a measure of organic carbon in a sample.

3.3 **BOD:** "Biochemical Oxygen Demand" is a measure of oxygen consumed by microorganisms under specific conditions.

4.0 Interferences

4.1 Organic contamination in the glassware or water used for dilutions will cause high results. Contamination of the calibration standards will cause low results. Even low concentrations of organic material will cause detectable errors.

4.2 Some inorganic compounds will contribute to COD. If COD is meant to be an indicator of organic contamination in these cases, the result will be too high. Reduced inorganic species such as ferrous iron, sulfide, and manganous manganese are oxidized quantitatively under the test conditions.

4.3 Pyridine and related compounds resist oxidation.

4.4 Volatile materials may be lost during sampling and in subsequent handling. Volatile organics are present in the vapor state under test conditions and do not come into contact with the oxidizing liquid.

4.5 Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentrations of free chloride ions.

4.6 Nitrite in concentrations of more than 2 mg/L is a positive interference.

4.7 Chloride is quantitatively oxidized by dichromate and will cause high results. Mercuric sulfate is present in the tubes to prevent interference from up to a maximum chloride concentration of 2000 mg/L. Samples containing more chloride than this must be diluted prior to analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The COD digestion tubes used in this analysis contain a proprietary mixture of compounds of mercury, silver, and chromium, which are highly toxic. Wear gloves and handle the tubes carefully. DO NOT POUR USED TUBES INTO SINKS. Take care not to overheat the tubes. This may cause them to leak or break. The block digester must be placed in a fume hood. The tubes will get very warm when sample is added. Carefully inspect tubes to ensure that they are not leaking before handling.

5.1.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4 The block digester is set to a maximum of 152 °C. Take care not to touch the hot surfaces directly to avoid burns.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

HACH COD Reagent	Corrosive Poison	0.1 mg/m^3 (Hg) 0.01 mg/m^3 (Ag) 1 mg/m^3 (H_2SO_4)	Toxic by inhalation. Causes severe burns and may cause difficult breathing, mouth soreness, and teeth erosion. Causes severe burns in contact with skin. Easily absorbed through the skin and may cause abdominal pain, circulatory disturbances, diarrhea, loosening of the teeth, nausea, vomiting, rapid pulse rate, toxic nephritis, shock, collapse, kidney damage, and death. Causes severe burns in contact with eyes.
1 – Always add water to acid to avoid violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Analytical balance capable of accurately weighing to the nearest 0.0001 g. Verify the accuracy of the balance each day it is used as described in SOP DV-QA-0014.
- Block digestor for 16 mm tubes, set at 150 ± 2 °C.
- Thermometer to measure block digestor temperature. Thermometers require periodic calibration checks against NIST thermometers as described in SOP DV-QA-0001.
- Spectrophotometer with adapter for 16 mm tubes, for absorbance measurements at 420 nm.

6.2 Supplies

- Volumetric Flasks (Class A): varying volumes.
- Eppendorf Pipettes, varying volumes
- Chloride test strips.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Premixed Low Level COD tubes, Hach (Method 8000) Corporation #21258-15.

7.2 Deionized Water

Deionized water must be free of significant organic carbon. Do not use water that has been stored in plastic squirt bottles; obtain fresh water from the tap. The TestAmerica Denver house deionized water meets the requirements of ASTM Type II water with a minimum resistivity of 10 Megohm-cm. Deionized water straight from the tap is used instead of ELGA water as the cartridges in the ELGA system may contribute small amounts of COD to the water.

7.3 COD Spike Solution, 10,000 mg/L

Dry reagent grade potassium hydrogen phthalate (KHP) to a constant weight at 120 °C. Dissolve 0.8503 g of KHP in water and dilute to 100 mL in a volumetric flask. If the final

concentration varies from 10,000 mg/L, subsequent dilutions must be adjusted accordingly. This solution may be purchased commercially.

7.4 COD Stock Standard, 1,000 mg/L

Use a certified standard purchased from a commercial source.

7.5 COD Working Calibration Standards

Dilute the 1,000 mg/L COD Stock Standard (Section 7.4) according to the table below to obtain the indicated concentrations. The working standards must be prepared fresh before each analysis.

Level	Volume of Stock (mL)	Final Volume (mL)	Final Conc (mg/L)
1	1.0	100	10
2	2.0	100	20
3	5.0	100	50
4	10	100	100
5	15	100	150
6	20	100	200

7.6 COD Laboratory Control Sample (LCS), 100 mg/L

The LCS is prepared in the same manner as the Level 4 calibration standard (100 mg/L) as described in Section 7.5.

7.7 COD Initial Calibration Verification (ICV) Stock Standard, 1000 mg/L

The ICV standard solution is a second-source standard that is obtained from a commercial source that is different from the source of the COD Stock Standard (Section 7.4)

7.8 Working ICV Standard, 100 mg/L

Dilute 10 mL of the 1000 mg/L ICV Stock Standard (Section 7.7) to 100 mL with deionized water.

7.9 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD), 50 mg/L

Matrix spikes are prepared by diluting 0.5 mL of the 10,000 mg/L COD Spike Solution (Section 7.3) to 100 mL with the sample selected for spiking.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Amber Glass	250 mLs	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

9.1.1 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.1.2 Method Blank

A method blank is required with every batch of 20 or fewer samples. The blank is deionized water taken through the procedure as if it were a sample.

Acceptance Criteria: The method blank must not contain COD above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be re-digested and reanalyzed

9.1.3 Laboratory Control Sample (LCS)

One LCS is required with each analytical batch. The LCS is prepared at a concentration of 100 mg/L as described in Section 7.6.

Acceptance Criteria: The recovery of the LCS must be within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean percent recovery, and must be no wider than 90 to 110 %.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be re-digested and reanalyzed

9.1.4 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD)

One MS/MSD pair is required for every 10 field samples. MS/MSDs are prepared to contain an additional 50 mg/L COD above the native COD concentration of the parent sample as described in Section 7.9.

Acceptance Criteria: The recovery of the MS and MSD must be within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean percent recovery, and must be no wider than 90 to 110 %.The relative percent difference (RPD) between the MS and MSD must be no greater than 20%.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.2 Instrument QC

All calibration standards, QC solutions, and field samples are analyzed using a 2 mL portion of sample that is digested.

9.2.1 Initial Calibration (ICAL)

The six calibration concentration levels 10 to 200 mg/L as shown in Section 7.5.

9.2.2 Initial Calibration Verification (ICV)

The second-source ICV standard (Section 7.8) is analyzed immediately following the ICAL.

Acceptance Criteria: The COD recovery in the ICV standard must be 90 - 110 %.

Corrective Action: If the recovery is outside of the acceptance limits, investigate the problem and repeat the ICAL. If the problem is traced to the digested standards, the entire batch must be re-prepared and reanalyzed.

9.2.3 Continuing Calibration Verification (CCV)

Analyze the CCV standard after every 10 or fewer samples and after the last sample. The CCV standard is prepared by digesting 2 mL of the 100 mg/L Level 4 calibration standard listed in Section 7.5.

Acceptance Criteria: The COD recovery in the CCV standard must be 90 - 110%.

Corrective Action: If the recovery is outside of the acceptance limits, repeat the ICAL and reanalyze all samples analyzed since the last successful CCV.

9.2.4 Continuing Calibration Blank (CCB)

Analyze a CCB is after each CCV. The CCB consists of 2 mL of deionized water that is digested and analyzed in the same manner as a sample.

Acceptance Criteria: The result for the CCB must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. Recalibrate the instrument and reanalyze all samples analyzed since the last successful CCV.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 All glassware must be thoroughly rinsed with deionized water to remove traces of detergents and other potential contaminants.

10.1.2 Turn on the block digestor and preheat to 150 ± 2 °C.

10.1.3 Prior to starting the digestion, screen all samples for chloride.

NOTE: The chloride screen may be omitted only when chloride data for the samples are available or when comparable results, such as TDS or conductivity, indicate it is not possible for the samples to contain > 2000 mg/L of chloride.

10.1.4 Use chloride test strips to screen for chloride. Record the chloride result on the bench sheet. Samples that are high in chloride are also noticeable for the large amount of precipitate that is formed when the sample is added to a COD tube.

10.1.5 Samples containing more than 2000 mg/L chloride must be diluted in order to bring the chloride concentration below this level. Do not over-

dilute the samples. Generate an NCM anomaly for any sample that requires dilution to reduce chloride interference.

10.1.5.1 When possible do not take sample aliquots for dilution that are less than 1.0 ml. It is preferable to take larger sample aliquots when possible to get a proper homogeneous sample.

Example: For a 2x dilution take 1 mL of your sample and dilute it to a final volume of 2 mL using DI water. Sample is ready for analysis

Example: For a 100x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. A 2 mL aliquot taken from the 100 mL is then used for analysis.

Standard COD Dilutions

Sample Aliquot	Final volume	Dilution
1.0 mL	2 mL	2x
1.0 mL	5 mL	5x
1.0 mL	10 mL	10x
1.0 mL	20 mL	20x
1.0 mL	50 mL	50x
1.0 mL	100 mL	100x

10.1.5.2 When samples require serial dilutions, typically greater than 100x, try to limit the amount of dilutions required to get to the final dilution.

Example: For a 1000x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. From the 100 mL take an aliquot of 1.0 mL and bring it up to a final volume of 10 mL. A 2 mL aliquot taken from the 10 mL is then used for analysis.

Example: For a 10,000x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. From the 100 mL take an aliquot of 1 and bring it up to a final volume of 100 mL. A 2 mL aliquot taken from the 100 mL is then used for analysis.

Standard Serial Dilutions

Sample Aliquot	Final Volume	Initial Dilution	Aliquot from initial dilution	Final Volume	Final Dilution
1.0 mL	100 mL	100x	1.0 mL	10 mL	1000x

1.0 mL	100 mL	100x	1.0 mL	20 mL	2000x
1.0 mL	100 mL	100x	1.0 mL	50 mL	5000x
1.0 mL	100 mL	100x	1.0 mL	100 mL	10,000x

10.1.6 Inspect each tube and discard any that are discolored. The presence of a precipitate in the tubes is normal.

10.1.7 Prepare the calibration standards for analysis by pipetting 2.0 mL of each standard solution into separate COD tubes.

10.1.8 Prepare the method blank by pipetting 2.0 mL of deionized water into a COD tube.

10.1.9 Prepare the LCS by pipetting 2.0 mL of the LCS solution (Section 7.6) into a COD tube.

10.1.10 Pipette 2.0 mL each of each field sample into separate COD tubes. Samples containing solid material should be homogenized first.

10.1.11 Using the sample selected for spiking, prepare the MS and MSD as described in Section 7.9, and pipette 2.0 mL of each into separate COD tubes.

10.1.12 Tighten the cap on each COD tube securely, but do not over-tighten. Label the tubes on the space provided with the appropriate identification.

10.1.13 Mix the contents in each tube by slowly inverting two or three times. At this point, the tube will become warm. If any of the tubes leak, the samples affected must be re-aliquotted.

10.1.14 Place the COD tubes in the heated block digestor for two hours and record the start time and the temperature on the bench sheet.

10.1.15 Remove the tubes from the block digestor after digesting for 2 hours and record the end time and temperature on the bench sheet.

10.1.16 Wait 20 minutes to allow the tubes to cool to 120 °C or less, then invert each tube several times while still warm. Place the tubes into a rack.

10.1.17 Wait until the tubes have cooled to room temperature before proceeding.

10.1.18 Inspect each tube and note any sample that shows signs of leakage (reduced volume, salts and/or charring on the outside of the tube). Re-digest any samples showing these signs.

10.1.19 Allow any precipitate in the samples to settle out fully before analysis.

10.2 Calibration

10.2.1 Calibrate the instrument using the six calibration concentration levels (10 to 200 mg/L as shown in Section 7.5).

NOTE: It is generally **NOT** acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest

or lowest on the curve **AND** the reporting limit and/or linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason is clearly documented, for example a cracked tube, and a minimum of five levels remain.

10.2.2 Digest and analyze the calibration standards in exactly the same manner as the samples as described in Section 10.1 and 10.3.

10.2.3 Use linear least squares regression to calculate a calibration function relating COD concentration to absorbance at 420 nm. Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (absorbance at 420 nm) versus the concentration of each standard. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

Equation 1

$$y = ax + b$$

Where:

y = Instrument response (absorbance at 420 nm).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

10.2.4 To calculate the concentration in an unknown sample, the regression equation is solved for concentration, resulting in the following equation, where x is now the concentration of the target analyte in the unknown sample extract:

Equation 2

$$x = \frac{y - b}{a}$$

10.2.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.2.6 **Calibration Acceptance Criteria**
The absolute value of the correlation coefficient for the linear equation must be 0.995 or greater. (Note that the correlation coefficient is always a negative value because of the inverse relationship between COD and chromate concentration.)

10.2.7 **Corrective Actions for Calibration Failures**
If the absolute value of the correlation coefficient is less than 0.995, the spectrophotometer conditions and calibration standards should be rechecked. Correct any problems found and recalibrate. Samples cannot be analyzed until the initial calibration is successful.

10.3 Sample Analysis

10.3.1 Turn on the spectrophotometer and allow it to warm up for 20 minutes.

10.3.2 Set the wavelength to 420 nm and zero using the Level 6 (200 mg/L) calibration standard.

NOTE: For clarification, any standard that is fully oxidized could be used to zero the spectrophotometer. The 200 mg/L standard is used for consistency and to fully challenge the oxidation step.

10.3.3 Wash each tube with water and dry using a Kimwipe before inserting it in the spectrophotometer. Any dirt or scratches on the tubes will affect results, so examine each tube carefully prior to analysis. Cover with the light shield while taking readings.

10.3.4 Measure the absorbance of each standard and sample. Record the absorbance to ± 0.001 units on the bench sheet.

10.3.5 Dilute and reanalyze any sample that has a result greater than 150 mg/L.

10.3.6 Turn off all equipment. Clean all glassware, apparatus, and the work area.

11.0 Calculations / Data Reduction

11.1 Concentration:

Enter the concentration and absorbance of each standard into a linear least squares regression program to generate the calibration function, as described in Section 10. The linear least squares regression equation (see Equation 1) is solved for concentration and results in the following equation that is used to calculate the COD concentration in the measured sample aliquots:

$$C = \frac{y - b}{a}$$

Equation 3

Where:

C = COD concentration in the measured sample (mg/L).

y = Instrument response for the sample (absorbance at 420 nm).

a = Slope of the line.

b =The y-intercept of the line.

11.2 Diluted Samples Concentration:

If the sample was diluted, multiply the calculated concentration by the appropriate dilution factor, as follows:

Equation 4

$$COD = C \times DF$$

Where:

C = COD concentration in the measured sample (mg/L).

DF = Dilution factor (DF is 1 if the sample was not diluted for measurement).

The reporting limit is 10 mg/L. Samples that have a COD concentration less than 10 mg/L are reported as not detected (ND). Samples that have a COD concentration greater than 150 mg/L must be diluted and reanalyzed.

Dilutions required for other reasons (e.g., high chloride, insufficient sample, or other interferences) must be explained in an NCM or other communication to the laboratory Project Manager to ensure that the analytical problem is explained in the final report case narrative.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD and AFCEE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 The initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

The digestion reagent used in this procedure is a powerful oxidizing reagent containing mercury. This procedure uses much less sample and digestion reagent, approximately one-twentieth as much, than the older macro-COD methods in order to minimize the use of mercury.

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Used or expired COD tubes containing reagent – COD Vials - Waste Stream I

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Methods for the Chemical Analysis of Water and Waste (MCAWW); EPA, 1983.

15.2 Method 410.4, The Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry, Revision 2.0, August 1993.

15.3 Standard Methods for the Examination of Water and Wastewater, 20th Edition; Clesceri, L.S.; Greenberg, A.E.; Eaton, A.D.; Editors; American Public Health Association, American Water Works Association, and Water Environment Federation, 1998.

16.0 Method Modifications:

Item	Method	Modification
1	410.4	The COD digestion tubes have been changed due to the instrument manufacturer's specifications. This change includes the use of premixed low level digestion tubes, which changes the sample volume added and the size of the digestion tube.
2	410.4	The EPA-approved Hach method 8000 is based on measurement of Cr+6 at 420 nm, rather than Cr+3 at 600 nm, which is used for EPA 410.4. A letter from EPA EMSL is on file, which verifies the acceptability of this approach.

17.0 Attachments

Attachment 1: Example COD Bench Sheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 6.3, dated 15 October 2010
  - Corrected LCS, MS and MSD acceptance criteria to 90-110% to meet method requirement.
-
- Revision 6.2, dated 02 March 2010
  - Annual Review
  - Added section 6.3

- Revision 6.1, dated 28 February 2009
  - Added Dilution tables in section 10.1
  - Added Hach method references

- Revision 6, dated 15 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Added the reference concerning not to use ELGA water in section 7.2.
  - Added location of MDLs to section 12.
  - Updated SOP references to the new naming convention. (ex. DEN-WC-0018 is now DV-WC-0018).
  - Added updated bench sheet and checklist with new logos.

- Revision 5, dated 24 July 2007
  - The reference to Standard Methods Method 5220D has been removed.
  - The company name has been changed from STL to TestAmerica.

- Revision 4, dated 14 November 2006
  - Updated formatting to be consistent with Policy QA-001.
  - Incorporated the Safety Bulletin to meet STL Corporate requirements.
  - Incorporated Interim Changes.
  - Expanded Section 4 to include interferences cited in the source methods.
  - Revised Section 7 to match current practice.
  - Deleted recording the digestion block temperature at startup (Section 10.1.2). The temperature is recorded when samples are placed in the block and when they are removed.
  - Added instruction to examine tubes prior to analysis for scratches, as well as dirt (Section 10.3.3).
  - Added clarifications to instructions for performing the chloride screen to Sections 10.1.3 through 10.1.5.
  - Revised Section 9.1 to include reference to Policy QA-024.
  - Added calibration equations to Sections 10 and 12.

- Revision 3, dated 29 July 2002
  - The summary of the method in Section 2 was expanded.
  - References to relevant QA SOPs and policies were added throughout.
  - The reference to the Chemical Hygiene Plan was changed to the Corporate Safety

Manual.
  - The calibration curve was changed to include a standard at 10 mg/L, the reporting limit.
  - Details concerning preparation of calibration standards and spike solutions were updated in Section 7.
  - QC requirements in Section 9 were clarified, and increased MS/MSD requirements for South Carolina and North Carolina added.
  - Calibration information and controls were moved from Section 9 to 10. Section 10 now includes warnings against removing calibration points. Section 10 also specifies linear regression with absolute value of correlation coefficient > 0.995.
  - Section 11.3.11 was edited to remove provision for rapid cooling of COD tubes.
  - The calculation formula was added to 12.4.
  - The Method Performance Section 13 was expanded to include requirements for MDLs, IDOCs, and training.
  - The Pollution Prevention Section 14 was expanded.
  - The Waste Management Section 15 now identifies the COD waste stream.
- Revision 2, dated 12 September 2000
  - The company name was changed from Quanterra to STL.
  - The lower reporting limit is changed from 5 mg/L to 10 mg/L.
  - The second source standard is identified as an ICV, rather than an LCS.
  - A clarification is made in section 10 for zeroing the instrument.
  - In Section 11.4.1 the samples needing dilutions are changed from 0.000 absorbance to greater than 150 mg/L.

- Revision 1, dated 20 May 1999
  - The benchsheet was amended such that more details regarding the calibration curve would be recorded and Quantims lot numbers and work order numbers would be recorded.
  - The upper limit of the working range was changed from 200 mg/L to 150 mg/L.
  - The reporting limit was revised from 20 mg/L to 10 mg/L.

Attachment 1.

Example COD Bench Sheet

Analyst:	KBERTHA	Calibration Curve Information						Instrument Information	
Date:	09/29/06		Conc.(mg/L)	ABS.	Conc.(mg/L)		ABS.		
QC Lot:		STD1	10	0.572				Instrument:	Spec 301
QC Run:		Std. 2	20	0.526				Wavelength:	420
DATE OF CURVE=	7/11/2006	Std. 3	50	0.441				Parameter:	COD
SOP Information		Std. 4	100	0.285				Corr. Coef:	-0.99947
Number:	Den-WC-0018	Std. 5	150	0.137	MDL =	4		Slope:	-0.00299
Revision:	1.0	STD 6	200	0	RL =	20		Intercept:	0.3913

ICV/ Information: TV = 100 mg/L		Calibration, LCS/Matrix Spike			
Source	Ricca	std #	1973-06	COD Tube Source:	HACH
lot#	1412298 STD 0662-06 NA	made by	HACH lot A5[illegible]6	Lot & Exp:	A5[illegible]6 30-Nov-10
Concentration:	1000mg/L	Concentration:	1,000 mg/L	Made By:	FISHER 10,000 mg/L
Expiration Date:	10/31/2006	Expiration Date	7[illegible]9/2007	STD3203-0[illegible]	lot 66686 exp: 12/[illegible]/2006
True value:	ICV True value: 100 mg/L	LCS	100 mg/L	MS/MSD	50 mg/L

Int./Notes	LIMS ID	CI	Sample Amount (mL)	Sample ABS.	Color Blank ABS.	Concentration (mg/L-mg/kg)	Prep D.F.	Anal D.F.	Final Conc. (mg/L-mg/kg)	% Rec.
12:00										
	ICV		2	0.288		101.30[illegible]15	1	1	101.30	101%
	ICB		2	0.606		-4.90462	1	1	ND	
	DCS		2	0.279		104.30903	1	1	104.31	104%
	DCS		2	0.267		108.31687	1	1	108.32	108%
	MB		2	0.606		-4.90462	1	1	ND	
JE793	D6I280181-1	>3000	2	0.470		40.[illegible]1757	1	10	405.18	
JE796	2	>3000	2	0.290		100.63518	1	5	503.18	
JE797	3	>3000	2	0.385		[illegible]8.90644	1	5	344.53	
JE799	4	>3000	2	0.396		6[illegible].23259	1	5	326.16	
JE8AA	5	>3000	2	0.422		56.[illegible]1893	1	5	282.74	
JE8AD	6	>3000	2	0.[illegible]89		0.773[illegible]5	1	5	ND	
JE8AE	7	>3000	2	0.425		56.21495	1	5	281.07	
	CCV		2	0.268		107.98288	1	1	107.98	108%
	CCB		2	0.604		-[illegible].23664	1	1	ND	
JE8AH	8	>3000	2	0.[illegible]90		[illegible]7.20[illegible]51	1	5	336.18	
JE8AK	9	<2000	2	0.606		-4.90462	1	1	ND	
JE71Q	D6I280148-2	<2000	2	0.595		-1.23076	1	1	ND	
MSD	2S		2	0.[illegible]29		50.87116	1	1	50.87	
MSD	2SD		2	0.[illegible]37		51.[illegible]3913	1	1	51.54	
JE711	3	<2000	2	0.59[illegible]		-0.562[illegible]	1	1	ND	
JE712	4	<2000	2	0.586		1.775[illegible]2	1	1	ND	
JE713	5	<2000	2	0.588		1.10[illegible]14	1	1	ND	
JE715	6	<2000	2	0.[illegible]00		[illegible].90070	1	1	ND	
JE717	7	<2000	2	0.58[illegible]		1.10714	1	1	ND	
	CCV		2	0.267		108.31687	1	1	108.32	108%
	CCB		2	0.602		-3.56867	1	1	ND	
JE72[illegible]	8	<2000	2	0.59[illegible]		-1.89874	1	1	ND	
JE722	9	<2000	2	0.589		0.77316	1	1	ND	
JE725	10	<2000	2	0.592		-0.22880	1	1	ND	
JE73F	12	<2000	2	0.[illegible]01		0.10518	1	1	ND	
	CCV		2	0.277		104.97700	1	1	104.98	105%
	CCB		2	0.602		-3.56867	1	1	ND	
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2				1	1		

Attachment 2.

Example Data Review Checklist

TESTAMERICA Denver

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RL / MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH checks documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Special client requirements met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples ≥ 20x blank have NCM?				
2. LCS run for batch and within QC limits ?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

7/6/07 Version
L:\QA\Edit\Forms\Wet Chemistry\Calib Curve Checklist

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Title: Biological Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD)

[SM 5210B / EPA 405.1]

Approvals (Signature/Date):

Dave Elkin — 12/21/10 Date
Wet Chemistry Supervisor

Adam Alban — 30 Dec 10 Date
Health & Safety Manager / Coordinator

John Morris — 12/30/10 Date
Quality Assurance Manager

Robert C. Hanisch — 12/30/10 Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is applicable to the determination of biochemical oxygen demand (BOD) by EPA Method 405.1 and Standard Method 5210 B, and carbonaceous biochemical oxygen demand (CBOD) by Standard Method 5210 B in waste water, effluents, polluted waters, and other aqueous samples.

1.1.2 This method is used to determine the relative oxygen requirements of wastewaters, effluents and polluted waters. The test allows calculation of the effect of organic waste discharges on the oxygen resources of the receiving water.

1.1.3 Under the conditions of this method, BOD or CBOD in the range of 2 to approximately 300 mg/L as dissolved oxygen (DO) can be determined. This can be extended to higher levels by dilution of the sample.

2.0 Summary of Method

2.1 The BOD test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The CBOD test is identical to the BOD test, except that a nitrification inhibitor is added.

2.2 A series of dilutions is performed on a sample with a nutrient buffer solution. The diluted samples are inoculated with an active microbial population and incubated in the dark at 20 °C for 5 days. The bottles used are sealed to prevent absorption of oxygen during the test.

2.3 The BOD (or CBOD, if nitrification inhibitor is used) of the sample is calculated from dissolved oxygen readings taken before and after the incubation period.

3.0 Definitions

3.1 **Biochemical Oxygen Demand (BOD):** BOD is an empirical test in which standardized laboratory procedures are used to determine the relative oxygen requirements of waste waters, effluents, and polluted waters. It is widely used for measuring waste loadings to treatment plants and for evaluating the BOD removal efficiency of such treatment systems. The BOD test measures oxygen consumed by microbial life while assimilating and oxidizing organic matter in the sample under standardized conditions of dark incubation at 20°C for 5 days. Actual environmental conditions of temperature, biological population, water movement, sunlight, and oxygen concentration cannot be reproduced in the lab.

3.2 BOD is one of several laboratory tests that can be used to characterize the type and extent of organic matter in water samples. Water samples can contain a variety of organic compounds in various states of oxidation. Some of these compounds can be further oxidized by **biochemical oxygen demand (BOD), assimilable oxygen demand (AOD), and chemical oxygen demand (COD).**

Another related analytical value is total organic carbon (TOC), which, as the name implies, measures the total amount of organic carbon in a water sample.

3.3 **Carbonaceous Biochemical Oxygen Demand (CBOD):** The CBOD test is identical to a BOD test, except that a nitrification inhibitor is added. If an inhibitor is not used, the BOD test will measure the sum of the carbonaceous and nitrogenous demands(from oxidation of reduced forms of nitrogen in water samples, such as ammonia and organic nitrogen).

4.0 Interferences

4.1 Variations in environmental conditions, such as temperature, biological population, water movement, sunlight, and oxygen concentration can affect BOD results. Conditions for the test have therefore been standardized to minimize these variations. Exposure to light may result in photosynthetic production of oxygen.

4.2 Bacterial growth requires nutrients such as trace metals, nitrogen, and phosphorus. These are added to the dilution water.

4.3 The pH must be in the range 6.5 to 7.5 to be suitable for the growth of bacteria.

4.4 Residual chlorine will kill microorganisms seeded into the sample. Chlorine must be destroyed before starting the analysis.

4.5 High concentrations of ammonia will contribute to BOD. This can be prevented by using a nitrification inhibitor, which results in a measurement of carbonaceous biochemical oxygen demand (CBOD). Inhibition of nitrification is recommended for samples of secondary effluent, for samples seeded with secondary effluent, and for samples of polluted water. It is the responsibility of the client to understand the nature of the water sample and to request the appropriate analytical method.

4.6 Organic contamination of glassware and reagents will cause high BOD results.

4.7 Oil and volatile organics in samples may cause an erroneous dissolved oxygen reading.

4.8 Some samples may contain compounds which are toxic to seed organisms. This is indicated by increasing BOD results with increasing dilution of the sample. Such samples may require special study and treatment.

4.9 Some compounds are not easily degraded by the seed organisms and will result in low BOD results.

4.10 Any factor which alters the dissolved oxygen concentration in a sample between the two readings will affect the BOD result. Avoid agitation of the sample. Take the initial dissolved oxygen reading as soon as possible after dilution, then stopper and seal the bottle. Some compounds will be oxidized in as little time as 15 minutes after dilution.

4.11 Due to possible contamination contributions the use of soap is prohibited in the washing of the dilution water reservoirs.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide (NaOH)	Corrosive Poison	2 mg/m3 (Ceiling)	Inhalation of dust or mist can cause a range of effects from mild irritation to serious damage of the upper respiratory tract. Symptoms may include sneezing, sore throat, or runny nose. Can cause irritation or severe burns and scarring in contact with skin. Causes irritation in contact with eyes and may cause burns that result in permanent impairment of vision, even blindness.
Sulfuric Acid (H2SO4)	Corrosive Water Reactive Poison Carcinogen	0.2 mg/m3 (TWA)	Inhalation damages mucous membranes and upper respiratory tract. Symptoms may include irritation of nose and throat and labored breathing. May cause lung edema. Can cause redness, pain, and severe burns in contact with skin. Prolonged skin contact can result in circulatory collapse with clammy skin, weak and rapid pulse, and shallow respirations. Can cause blurred vision, redness, pain, and severe tissue burns in contact with eyes. Chronic exposure to mists containing sulfuric acid is a cancer hazard.
Acetic Acid, Glacial	Corrosive Poison Flammable Liquid and Vapor	10 ppm (TWA)	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
1 - Always add acid to water to prevent violent reactions. 2 - Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Incubator controlled to 20 ± 1 °C. All light must be excluded. The incubator temperature is to be measured and recorded two times a day at least four hours apart. When the incubator temperature falls outside of 20 ± 1 °C, corrective action is taken as described in Section 10.3.15.1.
- A pH meter calibrated with a range covering 6.5 to 7.5. Narrow range pH paper covering the same range can be used.
- Dissolved oxygen meter. YSI Model 5100 with built-in barometric correction is currently in use, equivalent equipment can also be used. Consult manufacturer's instructions for proper use and storage of the probe. The probe is stored in a BOD bottle containing just enough water to cover the bottom.
- Magnetic stirrer, unless a self-stirring probe is used. Consult the manufacturer's instructions.
- Aquarium-type air pump, tubing, and bubbler.

NOTE: Please refer to the Master List of Documents and Software located on G:\QA\READ\Master List of Documents for the current software to be used for data processing.

6.2 Supplies

- BOD incubation bottles, 300 mL capacity, with ground glass stoppers and plastic caps. Disposable BOD bottles are purchased and are to be used one time only. If disposable bottles are not currently available, then glass BOD bottles will be used.
- Collapsible plastic bottles or carboys.
- Assorted laboratory glassware and apparatus such as pipettes, graduated cylinders, volumetric flasks, beakers, etc. All glassware must be scrupulously cleaned to remove traces of organic contaminants. For this reason, avoid using detergent to clean BOD bottles. Glass BOD bottles must be thoroughly cleaned with 10% HCl, rinsed with deionized water, and drained after cleaning.

7.0 Reagents and Standards

7.1 Reagent Water: Reagent water must be free of organics. Use Milli-Q water, distilled water, or other high purity water as available.

7.2 Chlorine Test Kit: Hach kit.

7.3 0.1N Sodium Hydroxide: Dissolve 0.4 g of sodium hydroxide in water, cool, and dilute to 100 mL.

7.4 **1.0N Sulfuric Acid:** Carefully add 2.8 mL of concentrated sulfuric acid to approximately 80 mL of deionized water, cool, and dilute to 100 mL with deionized water. This solution is usually purchased from Fisher.

7.5 **50% Glacial Acetic Acid:** Carefully add 10 mL of glacial acetic acid to 10 mL of deionized water.

7.6 **10% Potassium Iodide:** In a 100-mL volumetric flask, dissolve 10 g of potassium iodide (KI) in approximately 70mL of deionized water. Dilute to volume with deionized water.

7.7 **1% Starch Indicator Solution:** This reagent is available from commercial sources.

7.8 **Sodium Sulfite Solution:** Dissolve 0.16 g of sodium sulfite in deionized water and dilute to 100 mL with deionized water. This solution is unstable and must be prepared daily as needed. This solution may also be obtained from a commercial vendor.

7.9 **0.025 N Sodium Thiosulfate:** Dissolve 0.6205 g of sodium thiosulfate pentahydrate in deionized water and dilute to 100 mL with deionized water. This solution is unstable and must be prepared daily as needed. This solution may also be obtained from a commercial vendor.

7.10 **Dilution Water:** Add the contents of one HACH BOD Nutrient Buffer pillow to 3.0 liters of reagent water and aerate. It is recommended that dilution water older than 24 hours be used. Fresh dilution water may be used for analysis if necessary, but the dilution water blanks may be high due to small amounts of organic contamination in the reagents used. The dilution water is the first solution measured when setting up every BOD batch, and the quality check is explained in Section 10.3.3. The dilution water used in conjunction with this SOP is made daily.

7.11 **Seed (available commercially):** Add the contents of one Polyseed capsule to 500 mL of aerated dilution water and stir for one hour. Prepare fresh every day. Do NOT stir for longer than one hour. Always pre-test a new container of seed before use. This is done by adding the contents of one capsule to 500 mL of dilution water, and using the same aliquots per bottle, test the new seed along with the current seed as explained in Section 10.3.4. Increase or decrease the amount of dilution water if the new seed does not fall in the required range.

NOTE: Special projects may require the use of seed organisms adapted to the project waste stream.

7.12 **Nitrification Inhibitor (available commercially):** Dissolve 100 mg of 2-chloro-6-(trichloromethyl)pyridine in 100 mL of dilution water. Prepare fresh daily only if needed. Available commercially from HACH. Use two shots or 0.16 g per 300 mL bottle. This reagent is used for CBOD testing only.

7.13 **BOD LCS Solution (dextrose/glutamic acid):** Dry reagent grade dextrose and glutamic acid in a drying oven at 103 ± 5 oC. Cool in dessicator and store in a sealed container. In a 1-L volumetric flask, dissolve 150 mg of dextrose and 150

mg of glutamic acid in approximately 500 mL of deionized water and dilute to volume. This solution must be prepared fresh daily. Alternatively, a commercially-prepared dextrose/glutamic acid reagent may be purchased (packaged in ampules) from HACH or ERA. Dilute the reagents according to the manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass or Plastic	1 liter	Cool, 4°C /None	48 Hours	40 CFR Part 136.3

NOTE: The laboratory follows the 48 hour hold time as determined in the Standard Methods 20th edition QC section and 40 CFR Part 136.3.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.2 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.3 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.4 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.5 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.6 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	3 in 20 or fewer samples	198 ± 30.5 mg/L (85 to 115%)
Sample Duplicate[1]	1 in 20 or fewer samples	≤ 20% RPD
Dilution Water Blank	3 in 20 or fewer samples	< 0.2 mg/L of DO
Seed Control Samples	4 in 20 or fewer samples	The seed correction factor needs to be between 0.6 and 1.0 mg/L

[1]The sample selection for the sample duplicate is randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

9.7 Instrument QC

9.7.1 Some regulatory programs require that the meter be calibrated versus the Winkler titration. See Attachment 7.

9.7.2 Check the probe calibration as indicated on the bench sheet. The first probe check is done at the start of the test, before any samples, including the blank and the LCS, have been read. Subsequent probe checks are done after every 9 readings and at the end of the run.

Acceptance Criteria: Probe calibration checks must be within 2% of the expected value. Allow sufficient time for stabilization

Corrective Action: Adjust calibration if necessary and repeat all readings following the last acceptable probe check. If the probe will not calibrate properly, it may be necessary to change the membrane. Consult the manufacturer's instructions.

9.7.3 The probe is usually calibrated in water-saturated air, but can also be water calibrated. The procedure for this depends on the model of dissolved oxygen meter being used. Attachment 1 and Attachment 2 give the instructions for probe calibration of the Orion Dissolved Oxygen Probe and YSI Model 5100 Probe, respectively.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Precautions:

10.1.1.1 All additions of sample, reagents, dilution water, and seed must be made in such a way as to avoid entrapment of air.

10.1.1.2 Air bubbles must be excluded from the BOD bottles when making dissolved oxygen readings and again when placing stoppers on the bottles. Tapping on the side of the bottle can help in freeing any trapped air bubbles.

10.1.1.3 Dissolved oxygen readings must be taken IMMEDIATELY after making a dilution. Do not prepare several samples and then measure the dissolved oxygen. Dilute and measure each sample individually.

10.1.1.4 A water seal must be formed on top of the stoppers to prevent evaporation and entry of air. A plastic cap is used to prevent evaporation of the water seal.

10.1.1.5 Due to possible contamination contributions the use of soap is **prohibited** in the washing of the dilution water reservoirs. Reservoirs should only be cleaned with repeated rinses of DI water. A brush can be used to remove algae.

10.2 Calibration

10.2.1 Some regulatory programs require that the meter be calibrated versus the Winkler titration. See Attachment 7.

10.2.2 Check the probe calibration as indicated on the bench sheet. The first probe check is done at the start of the test, before any samples, including the blank and the LCS, have been read. Subsequent probe checks are done after every 9 readings and at the end of the run.

Acceptance Criteria: Probe calibration checks must be within 2% of the expected value. Allow sufficient time for stabilization.

Corrective Action: Adjust calibration if necessary and repeat all readings following the last acceptable probe check. If the probe will not calibrate properly, it may be necessary to change the membrane. Consult the manufacturer's instructions.

10.2.3 The probe is usually calibrated in water-saturated air, but can also be water calibrated. The procedure for this depends on the model of dissolved oxygen meter being used. Attachment 1 and Attachment 2 give the instructions for probe calibration of the Orion Dissolved Oxygen Probe and YSI Model 5100 Probe, respectively.

10.3 Sample Analysis

10.3.1 Measure the pH of each sample with a pH meter and record the value. If necessary, adjust the pH of an aliquot (approximately 500 mL) of each sample to between 6.5 and 7.5 by drop-wise addition of 0.1 N sodium

hydroxide or 1 N sulfuric acid, as appropriate. Do not use more than 1% of total sample volume of NaOH or H2SO4 to adjust pH.

10.3.2 Check for residual chlorine using the chlorine test kit.

10.3.2.1 Pour out approximately 10 mL of sample into a small container and add the chlorine test kit reagent.

10.3.2.2 If the solution turns pink, the sample contains residual chlorine and must be dechlorinated.

10.3.2.3 Remove residual chlorine by adding either sodium sulfite solution or sodium thiosulfate solution. The addition of sodium sulfite is done drop wise while stirring until the sample no longer tests positive for chlorine.

10.3.2.4 Recheck the pH, as sodium sulfite addition will increase the pH. Record the results of the test (positive or negative) on the bench sheet. See Attachment 5 Chlorine Neutralization Chart.

10.3.3 Dilution Water Check Samples (3 required).

10.3.3.1 Fill a BOD bottle with dilution water to just inside the neck.

10.3.3.2 Immediately measure and record the initial dissolved oxygen (DO) reading and the bottle number.

10.3.3.3 The initial DO reading of the dilution water should be between 6.5 and 9.0 mg/L depending on the elevation. It should not exceed 9.0 mg/L. If the dilution water is not within this range, allow the water to reach room temperature in a partially filled container and shake vigorously or aerate. Proper aeration is usually achieved with an aquarium air pump in 45 minutes to 1 hour.

10.3.3.4 Stopper the bottle, taking care to exclude all air bubbles. Be sure that there is a small amount of excess water on top of the stopper to form a water seal.

10.3.3.5 Cover the stopper with a plastic cap to prevent evaporation of the water seal and incubate with the samples.

10.3.4 Seed Controls

10.3.4.1 Be sure the seed is well mixed. Aliquot 15, 20, 25, and 30 mL of seed into 4 BOD bottles and fill to just inside the neck with dilution water.

10.3.4.2 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.4.3 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.5 Method Blank (MB)

10.3.5.1 Aliquot 4.0 mL of seed into a BOD bottle and fill to just inside the neck with dilution water (approximately 300 mL of dilution water).

10.3.5.2 For a CBOD method blank, aliquot 4.0 mL of seed into a BOD bottle, add 0.16 g or two shots of nitrification inhibitor, and fill to just inside the neck with dilution water.

10.3.5.3 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.5.4 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.6 Laboratory Control Sample (LCS)

10.3.6.1 Add 4.0 mL of well-mixed seed into each of 3 BOD bottles.

Note: If CBOD is required add 2 shots or 0.16 gram of the nitrification inhibitor to the three LCSs.

10.3.6.2 Pipette 6 mL of LCS (7.13)standard solution and dilute to just inside the neck with dilution water.

10.3.6.3 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.6.4 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.6.5 The final concentration of the LCS should be 198 mg/L +/- 30 mg/L. Be careful not to contaminate the stock solution with seed or any other material.

10.3.7 For each sample, pipette 4.0 mL of seed into a BOD bottle. If nitrification inhibitor is required (CBOD), add 2 shots or 0.16 gram of the nitrification inhibitor and note the addition on the bench sheet and anomaly sheet.

10.3.8 Add the sample aliquot (see below), and dilute to just inside the neck with dilution water.

10.3.9 Immediately record the initial dissolved oxygen reading, the sample volume used and the bottle number.

10.3.10 The initial DO reading must be less than 9.0 mg/L and greater than 6.5 mg/L. If it is not, dilute the sample until a suitable reading is obtained. Do not aerate samples unless requested by client, as this could cause loss of volatiles.

10.3.11 Stopper the bottle and form a water seal. Cover with a plastic cap and incubate for 5 days.

10.3.12 If historical data are not available, use the following guidelines for determining sample dilutions:

- 0.0 to 1.0% for strong industrial wastes,
- to 5% for raw and settled wastewater,

- 5 to 25% for biologically treated effluent, and
- 25 to 100% for polluted river waters.

NOTE: If historical data is not available a minimum of five aliquots **must** be set.

10.3.13 A COD screen may also be performed to assist in determining the appropriate dilution for BOD analysis when historical data are variable or unavailable. The COD screen is performed using the reagents and general procedures presented in SOP DV-WC-0018. For screening purposes, it is not necessary to comply with all the QC requirements stipulated in the SOP, as the results will not be reported.

10.3.13.1 The approximate highest value of BOD that could be expected in the sample is estimated by multiplying the COD screening result by 0.6.

10.3.13.2 Using the table below, determine which dilution would be appropriate for the estimated highest BOD value so as to choose the most concentrated aliquot that will be analyzed.

10.3.13.3 Then choose several more dilute aliquots (4 to 6 depending on how much historical data are available for the sample) in a geometric progression so that the most likely BOD values are covered.

Aliquot (mL)	Approximate BOD Range (mg/L)
240	2 - 6
120	5 - 13
60	10 - 25
25	24 - 60
10	60 - 150
5	120 - 300

NOTE: Color and/or odor may be used as a guide in determining which dilutions to make. Raw wastes and highly polluted waters may require an initial dilution (see next paragraph).

10.3.13.4 Do not take aliquots less than 1 mL. If a sample requires larger dilutions, perform an initial 5X (or higher) dilution with dilution water, then take the required aliquots. The range will be extended by the initial dilution factor.

10.3.14 Check the probe calibration every 9 samples as indicated on the bench sheet and at the end of the batch.

10.3.15 Place BOD bottles into the incubator and incubate samples for for 5 days ± 2 hours. Label the first bottle in the batch with the date and approximate time that the batch is to be read back.

10.3.15.1 If during the 5-day incubation period, the temperature falls outside of the acceptable range, i.e., 20 ± 1 °C, record the temperature excursion on an NCM.

10.3.16 Final Dissolved Oxygen Readings

10.3.16.1 After 5 days (± 2 hours), remove the BOD bottles from the incubator.

10.3.16.2 Calibrate the probe and measure and record the final dissolved oxygen in each bottle. Check the probe calibration as indicated on the bench sheet(every 9 samples and at the end of the run)

10.3.17 Clean all glassware, apparatus, and the work area.

11.0 Calculations / Data Reduction

11.1 Dilution Water Samples:

$$DOU = DO1 - DO2$$

Where:

DOU = Dissolved oxygen uptake, mg/L
DO1 = Initial dissolved oxygen, mg/L
DO2 = Final dissolved oxygen, mg/L

11.2 Seed Correction: (depletion must be 40 – 70% to be acceptable)

$$SEED = DOUSEED \times F$$

Where:

SEED = Seed correction, mg/L (must be between 0.6 and 1.0 mg/L)
DOUSEED = DO1 - DO2

Where:

DO1 = Initial dissolved oxygen, mg/L and
DO2 = Final dissolved oxygen, mg/L (should read ≥ 1.0 mg/L)

$$F = \frac{\%\ \text{seed in each BOD sample}}{\%\ \text{seed in control bottle}}$$

NOTE: Typically 4 mL of seed is used, which makes the numerator above = 1.33%.

11.3 Samples and LCS

11.3.1 Reject all sample aliquots with DO2 < 1 mg/L.

11.3.2 Reject all sample aliquots with (DO1 - DO2) < 2 mg/L.

11.3.3 For all others, calculate BOD as follows:BOD = (DO1 - DO2 - SEED) x (300/V)

Where: BOD = BOD for sample aliquot, mg/L.
DO1 = Initial dissolved oxygen, mg/L.
DO2 = Final dissolved oxygen, mg/L.
SEED = Seed correction calculated above (Section 11.2), mg/L.
V = Sample aliquot volume, mL.

11.3.4 Calculate and report the average BOD for all sample aliquots which meet the two criteria above, unless there is evidence of toxicity or another anomaly. Consult the group leader or senior analyst in such a case.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

12.2 Initial Demonstration of Capability

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample (e.g., LCS) containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria (e.g., LCS control limits). All four results must meet acceptance criteria before the method can be used to analyze samples.

12.2.4 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 successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and

has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Treated Sample Waste – Neutral - Waste Stream W

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method 405.1, "Biochemical Oxygen Demand", USEPA, Editorial Revision, 1974.

15.2 Method 5210B, "5-Day BOD Test", Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

15.3 Related SOPs

15.3.1 DV-WC-0018, Chemical Oxygen Demand

15.3.2 DV-WC-0006, Carbon In Water (TOC, TIC, DOC, and TC)

16.0 Method Modifications:

Item	Method	Modification
1	EPA 405.1: Section 3.1	The probe method is used for the dissolved oxygen measurements. It is NOT calibrated versus the Winkler method unless otherwise stated on the bench sheet.
2	SM 5210B: Section 4E.2	The reference method calls for residual chlorine to be determined by titration on one portion of the sample and a second portion treated for chlorine removal. This is not done since there is usually not enough sample. Instead, an aliquot of the sample is treated until it gives a negative test for chlorine.
3	SM 5210B: Section 4A	A pre-packaged nutrient buffer is used in place of the four individual solutions specified in the method. The final dilution water has the same composition.
4	SM 5210B: Section 4D.1	Pre-packaged lyophilized seed is used since it is not always possible to obtain seed from the waste treatment plant on evenings and weekends. This is an acceptable option in Method 5210B. The proportion of seed used is in accordance with the manufacturer's instructions.
5	SM 5210B: Secttion 6B	Due to the altitude of the laboratory facility, the acceptable initial DO level has been lowered to 6.5 from the recommended 7.0.

17.0 Attachments

Attachment 1: Instructions for Orion Dissolved Oxygen Probe

Attachment 2: Instruction for YSI Model 5100 Dissolved Oxygen Meter

Attachment 3: Example BOD Cover Sheet (i.e., data review checklist)

Attachment 4: Flow Chart

Attachment 5: Pre-treatment for the Presence of Chlorine

Attachment 6: Winkler DO Method and Sodium Thiosulfate Standardization Procedure

Attachment 7: Temperature – Dissolved Oxygen Calibration Values Table (5340ft altitude)

Attachment 8: Calibration Values for Various Atmospheric Pressures and Altitudes

Attachment 9: YSI Model 5100 Barometer Calibration Instructions

18.0 Revision History

- Revision 8 dated 30 December 2010
  - Added Attachments 7, 8 and 9.
  - Deleted Attachment 3

Revision 7 dated 02 October 2009

  - Added note to section 6.1 – Master List of Documents
  - Deleted the sentence concerning the washing of glass bottles in section 6.2.
  - Modified sections 10.3.3.3 and 10.3.10 to have the minimum DO reading 6.5.
  - Added Method Modification #5 to section 16.0.
  - Updated Attachment 4 to current checklist.
- Revision 6.1 dated 27 May 2009
  - Updated minor grammatical and formatting errors.
  - Added note concerning hold time to section 8.
  - Updated section 10.3.2 to match current practices.
  - Added note to section 10.3.12 concerning the use of 5 aliquots for samples without historical data.
  - Updated Attachment 4.

- Revision 6 dated 27 May 2008
  - Revised formatting throughout the SOP to be consistent with the new corporate guidance.
  - Added section 4.11 –prohibition of soap.
  - Additional information added to the section 5.
  - Added section 10.1.15 – prohibition of soap.
  - Added note in section 10.3.6 concerning nitrification inhibitor added to LCSs.

- Revision 5 dated 30 November 2007
  - Integration for TestAmerica and STL operations.
  - Revised formatting throughout the SOP to be consistent with the new corporate guidance.
  - Changed 10.2.2 to reflect check done at end of run.

- Revision 4 dated 28 June 2006
  - Revised formatting throughout the SOP to be consistent with the guidance in Policy QA-001.
  - Section 5, Safety, was revised to meet current STL requirements.
  - Added pH meter to Section 6 and deleted pH paper from Section 7.
  - Incorporated Interim Change to add the chlorine test kit to Section 7.
  - Revised Section 13, Pollution Prevention, to meet current STL requirements.
  - Added Section 15, Waste Management, to meet current STL requirements.

- Revision 3 dated 16 August 2002
  - References to Quanterra were changed to STL.
  - Reference to the Chemical Hygiene Plan was changed to the Corporate Safety Manual.
  - Reagents 7.6, 7.7, 7.8 added for the residual chlorine removal
  - Section 6.3 was changed to require two temperature checks daily for the incubator.

- Section 7.10 was changed so that the glucose/glutamic acid is prepared fresh daily.
- Sections 7.6, 7.7 and 7.8 were added as the reagents for residual chlorine removal procedure.
- Section 11.4.3 was updated to add titration to the residual chlorine removal procedure.
- Sections 11.5.1.3 and 11.5.4 were changed so that the initial DO requirement is 7.0 to 9.0 mg/L.
- Waste management section updated.

Attachment 1.

Instructions for Orion Dissolved Oxygen Probe

NOTE: The overflow funnel must be substantially modified in order to work with 60 mL BOD bottles. Be sure that it forms a tight fit in the neck of the bottle.

NOTE: Do not allow the membrane to become scratched or damaged in any way, and do not touch it with your fingers.

1. Calibration

- Connect the probe to the meter. Turn the meter to pH mode and the probe to OFF. Use the meter calibration knob to adjust the reading to 7.00.
- Turn the probe switch to BATT. The reading should be greater than 13 or off-scale. If not, the batteries need to be replaced.
- Turn the probe to ZERO. Use the calibration knob on the left to adjust the reading to 0.00.
- Insert the probe into a BOD bottle containing enough water to cover the bottom of the bottle. **THE PROBE TIP MUST NOT BE IMMERSED IN WATER OR HAVE WATER DROPLETS CLINGING TO THE MEMBRANE.** Gently touch the corner of a Kim-wipe to any water drops to very carefully dry the membrane if necessary. This bottle is also used for storing the probe.
- Turn the probe to AIR, allow to stabilize and use the calibration knob on the right to adjust the reading to the proper calibration value. This value depends on altitude and at TestAmerica Denver, it is 6.28. The value can be obtained from the table in the manufacturer's instruction book. Record this value on the bench sheet.
- Turn the probe switch to H2O for measurement of samples.

2. Measurement of Dissolved Oxygen

- Be sure that the probe has been set to H2O.
- Place the sample to be measured on a magnetic stirrer. Be sure that the stirrer is rotating.
- Wait for the reading to stabilize, then record it.
- Rinse the probe tip with a stream of deionized water or dilution water between measurements.
- Store the probe in a BOD bottle with a small amount of water in the bottom and turn it off when not in use. Do not store the probe with the tip completely immersed in water.

Attachment 1.

Instructions for Orion Dissolved Oxygen Probe (Continued)

3. **Calibration Check**

- Carefully blot and dry off the tip of the probe by touching the corner of a Kim-wipe to any water droplets. Do not damage or touch the membrane with your fingers.
- Place the probe in a BOD bottle containing a small amount of water in the bottom. Do not immerse the membrane.
- Switch the probe to the AIR position and observe the reading.
- When the reading stabilizes, it should be between 6.15 and 6.41.
- Return the probe to H2O before continuing with any more dissolved oxygen measurements.etc....

Attachment 2.

Instruction for YSI Model 5100 Dissolved Oxygen Meter

NOTE: Do not allow the probe to become scratched or damaged in any way, and don't touch it with your fingers.

1. **Calibration**

- Place the probe in a BOD bottle with a small amount of water in the bottom.
- Do not immerse the membrane.
- Turn the function switch to TEMP oC. A tone will sound followed several seconds later by a second tone.
- Allow the meter to warm up for 15 minutes. The warm-up time may be eliminated if the probe is not turned off after use.
- Record the temperature of the probe on the bench sheet.
- Determine the calibration value for the probe temperature from the table below (these values have already been corrected for TestAmerica-Denver's altitude; consult the manufacturer's instruction manual for corrections at other altitudes/pressures). Record this value on the bench sheet.
- Set the switch to mg/L CAL and use the keypads beneath the display to set the calibration value obtained in step 1.5.
- Turn the switch to mg/L. CAL will appear in the display, followed in a few seconds by a tone. The reading will then be displayed; it should match the calibration value.
- Observe the reading for 2 to 3 minutes. If it drifts by more than 0.02 units, insufficient warm-up was allowed. Repeat steps 1.4 through 1.7, if necessary. Otherwise, the probe is now calibrated and ready for use.

2. **Measurement of Dissolved Oxygen**

- Place the probe into the sample to be measured and turn on the stirrer motor.
- Record the reading when stable.
- Turn off the stirrer and remove the probe from the bottle.
- Rinse the probe with a stream of water after each measurement.
- Store the probe in a BOD bottle containing a small amount of water. Do not immerse the membrane.

Attachment 2.

Instruction for YSI Model 50 Dissolved Oxygen Meter (continued)

3. **Calibration Check**

- Place the probe in a BOD bottle with a small amount of water in the bottom.
- Record the reading when stable.
- Switch back to TEMP oC and record the temperature.
- Look up the calibration value for this temperature from the table.
- The reading should match the calibration value within 2%.
- Switch back to mg/L and continue with readings.

YSI Model 50 Dissolved Oxygen Meter
Calibration Values (for TestAmerica Denver altitude)

Temperature (°C)	Calibration Value		Temperature (°C)	Calibration Value
15	8.28		23	7.05
16	8.11		24	6.92
17	7.94		25	6.79
18	7.78		26	6.70
19	7.62		27	6.32
20	7.47		28	6.43
21	7.33		29	6.32
22	7.18		30	6.21

Attachment 3.

Example BOD Data Review Checklist

TestAmerica Denver

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BOD Data Review Checklist

Methods: EPA 405.1, Standard Methods 5210 B

SOP # DV-WC-0019

Run Date:_____ **Analyst:**_____ **Instrument:**_____

Log-in / Sample Numbers	Batch	Method	Special Inst

A. Materials and Instrument Checks	Yes	No	N/A	2nd Level
1. Seed water stirred/aerated for at least one hour?				
2. Incubator temperature in control (20 ± 1°C) for 5-day incubation?				
3. Probe checks done at prescribed intervals as shown on the benchsheet?				
4. If CBOD in batch, is there a Blank, Duplicate for CBOD in the batch?				
B. Method Required QC				
1. Initial D.O. readings for samples, blanks and GGA standard between 7.0 and 9.0 mg/L?				
2. Seed control depletion is at least 2.0 mg/L (40 – 70%)?				
3. Seed correction value between 0.6 and 1.0 mg/L?				
4. Glucose/Glutamic acid (LCS) within control limits?				
C. Sample Results				
1. 48 hour holding time met for all samples?				
2. Was sample pH between 6.5 and 7.5 (adjustment made if necessary)?				
3. Were samples checked for residual chlorine (removed if necessary)?				
4. Are all sample dilutions with depletion <2.0 mg/L, or final D.O. <1.0 mg/L rejected?				
5. Do reporting limits reflect dilutions and/or limited sample volume?				
6. Were there any signs of toxicity in the samples?				
7. Were there any air bubbles in the BOD bottles?				
8. Were special client requirements met?				
9. STD/True Value information is updated and included?				

Analyst: _____ **Date:** _____

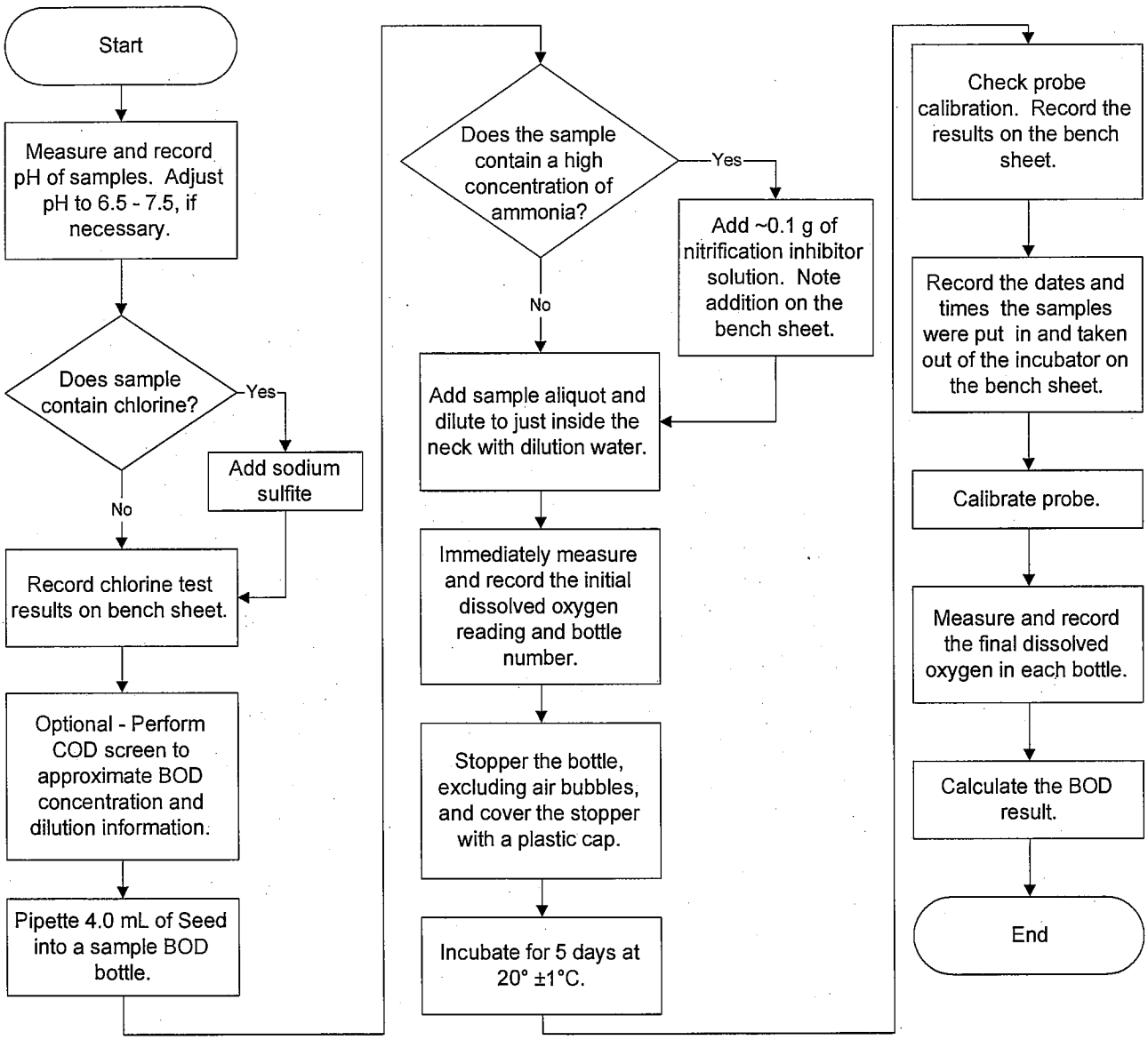
_____

_____

2nd Level Reviewer : _____ **Date:** _____

Attachment 4.

Flow Chart



Attachment 5.

Chlorine Neutralization Chart[1]

Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)
0.05	0.10	2.10	4.20	4.15	8.40	6.20	12.5
0.10	0.20	2.15	4.30	4.20	8.50	6.25	12.6
0.15	0.30	2.20	4.40	4.25	8.60	6.30	12.7
0.20	0.40	2.25	4.50	4.30	8.70	6.35	12.8
0.25	0.50	2.30	4.60	4.35	8.80	6.40	12.9
0.30	0.60	2.35	4.70	4.40	**8.90**	**6.45**	13.1
0.35	0.70	2.40	4.80	4.45	9.00	6.50	13.2
0.40	0.80	2.45	4.90	4.50	9.10	6.55	13.3
0.45	0.90	2.50	5.00	4.55	9.20	6.60	13.4
0.50	1.00	2.55	5.10	4.60	9.30	6.65	13.5
0.55	1.10	2.60	5.20	4.65	9.40	6.70	13.6
0.60	1.20	2.65	5.30	4.70	9.50	6.75	13.7
0.65	1.30	2.70	5.40	4.75	9.60	6.80	13.8
0.70	1.40	2.75	5.50	4.80	9.70	6.85	13.9
0.75	1.50	2.80	5.60	4.85	9.80	6.90	14.0
0.80	1.60	2.85	5.70	4.90	9.90	6.95	14.1
0.85	1.70	2.90	5.80	4.95	10.0	7.00	14.2
0.90	1.80	2.95	5.90	5.00	10.1	7.05	14.3
0.95	1.90	3.00	6.00	5.05	10.2	7.10	14.4
1.00	2.00	3.05	6.10	5.10	10.3	7.15	14.5
1.05	2.10	3.10	6.20	5.15	10.4	7.20	14.6
1.10	2.20	3.15	6.30	5.20	10.5	7.25	14.7
1.15	2.30	3.20	6.40	5.25	10.6	7.30	14.8
1.20	2.40	**3.25**	**6.60**	5.30	10.7	7.35	14.9
1.25	2.50	3.30	6.70	5.35	10.8	7.40	15.0
1.30	2.60	3.35	6.80	5.40	10.9	7.45	15.1
1.35	2.70	3.40	6.90	5.45	11.0	7.50	15.2
1.40	2.80	3.45	7.00	5.50	11.1	7.55	15.3
1.45	2.90	3.50	7.10	5.55	11.2	7.60	15.4
1.50	3.00	3.55	7.20	5.60	11.3	7.65	15.5
1.55	3.10	3.60	7.30	5.65	11.4	7.70	15.6
1.60	3.20	3.65	7.40	5.70	11.5	7.75	15.7
1.65	3.30	3.70	7.50	5.75	11.6	7.80	15.8
1.70	3.40	3.75	7.60	5.80	11.7	7.85	15.9
1.75	3.50	3.80	7.70	5.85	11.8	7.90	16.0

Attachment 5.

Chlorine Neutralization Chart[1] (continued)

Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)
1.80	3.60	3.85	7.80	5.90	11.9	7.95	16.1
1.85	3.70	3.90	7.90	5.95	12.0	8.00	16.2
1.90	3.80	3.95	8.00	6.00	12.1	8.05	16.3
1.95	3.90	4.00	8.10	6.05	12.2	8.10	16.4
2.00	4.00	4.05	8.20	6.10	12.3	8.15	16.5
2.05	4.10	4.10	8.30	6.15	12.4	8.20	16.6
8.25	16.7	8.55	17.3	8.85	17.9	9.15	18.5
8.30	16.8	8.60	17.4	8.90	18.0	9.20	18.6
8.35	16.9	8.65	17.5	8.95	18.1	9.25	18.7
8.40	17.0	8.70	17.6	9.00	18.2	9.30	18.8
8.45	17.1	8.75	17.7	9.05	18.3	9.35	18.9
8.50	17.2	8.80	17.8	9.10	18.4	9.40	19.0

[1] The concentration of residual chlorine present must first be determined, refer to section 10.3.2. This chart shows the amount of 0.025 *N* sodium sulfite to be added to a 2-liter sample aliquot with the applicable concentration of residual chlorine. The amount of sodium sulfite that is added to the sample aliquot must be adjusted, unless a 2-liter aliquot is used. To determine the amount of sodium sulfite to be added to the sample aliquot, divide 2-liters by the volume of sample (in liters) being adjusted, then divide that factor into the amount of sodium sulfite to be added listed in the chart.

It is important that this chart is followed as closely as possible to avoid over de-chlorination. Excess amounts of sodium sulfite can add a positive bias to the final BOD result. Also, incomplete de-chlorination can lead to low BOD results since chlorine can kill seed organisms thus reducing the amount of oxygen depletion of the sample during incubation.

Do not add more than 1% of total volume of 0.025*N* Na_2SO_3. If a greater volume of Na_2SO_3 is required to de-chlorinate the sample, use a higher concentration of Na_2SO_3.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure

Winkler DO Method

This method is used to measure the DO content of the dilution water.

1. To the BOD bottle, add 2 mL of manganous sulfate solution by holding the tip of the pipet just above the surface of the water.
2. Add 2 mL of alkaline-iodine-azide reagent by placing the tip of the pipet just under the surface of the water.
3. **IMMEDIATELY** re-stopper the bottle so that no air bubbles are trapped inside.
4. Mix gently by inverting the bottle fifteen times to form a manganese hydroxide flock that is brown and cloudy.

NOTE: If no DO is present in the bottle, the flock will appear as a white, cloudy substance. **REGARDLESS OF WHAT COLOR APPEARS, CONTINUE TO THE ACID ADDITION IN STEP #5 BELOW.**

5. Allow the flock to settle until a clear supernatant appears in at least half of the bottle.
6. Re-mix as in step #4 above, and allow to settle again.
7. Remove the stopper and add 2 mL of concentrated sulfuric acid by holding the tip of the pipet just **above** the surface of the water.
8. Re-stopper the bottle so no air bubbles are trapped inside.
9. Mix gently by inverting the bottle fifteen times.

NOTE: If this produces a clear, colorless solution in the bottle, **the DO must be reported as <1 mg/L.**

10. After the acid is added, do not delay more than 45 minutes to perform the titration.
11. If color is present in the bottle, transfer the entire contents of the bottle to a 500 mL Erlenmeyer flask.
12. While stirring, titrate using a buret filled with 0.037*N* sodium thiosulfate standard titrant until a pale, straw color remains.
13. Continue stirring, and add 2 mL of starch indicator solution, which will turn the water, a blue color. Continue stirring and titrating slowly until a clear end point is achieved.

NOTE: DISREGARD ANY RETURN OF THE BLUE COLOR.

14. Record the volume of the titrant used.
15. If the normality of the sodium thiosulfate standard titrant is within the specified range as determined in the Normality Standardization Procedure, the 1 mL of titrant is equal to 1 mg/L of DO.
16. If not, apply the factor to determine the DO.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure (continued)

Sodium Thiosulfate Standardization Procedure

This method is used to determine the normality of the sodium thiosulfate titrant used in the Winkler DO procedure.

1. Add 150 mL of distilled water to a 350 mL Erlenmeyer flask.
2. Add approximately 2 grams of KI crystals and mix until dissolved.
3. Add 10 mL of 10% sulfuric acid solution.
4. Add 20 mL of 0.0375*N* potassium bi-iodate standard and mix.
5. Place in the dark for 5 minutes.
6. Remove from the dark, dilute to 300 mL with distilled water, and mix.
7. While stirring the standard, fill a buret with the 0.0375*N* sodium thiosulfate standard titrant and titrate to a pale straw color.
8. Continue stirring, and add 2 mL of starch indicator solution.
9. Continue to titrate until a clear end point is reached and disregard the return of any blue color.
10. Record the amount of titrant used and calculate the normality of the sodium thiosulfate standard titrant using the following formula:

$$\textbf{Volume}_1 \textbf{ x Normality}_1 = \textbf{Volume}_2 \textbf{ x Normality}_2$$

If the normalities are not equal, a 1-to-1 relationship is not present and 1 mL of titrant will not be equal to 1 mg of DO in the sample. A correction factor must be determined by dividing the calculated sodium thiosulfate normality by the known bi-iodate normality. Multiply this factor by the volume of titrant used in each Winkler DO to determine the amount of DO in each sample.

The following is an example calculation:

- For this example, the normality of potassium bi-iodate is 0.0375*N*, 20 mL of potassium bi-iodate was used, and 18.6 mL of sodium thiosulfate titrant was used.

$$V_1N_1 = V_2N_2$$
$$20.0 \text{ mL} \times 0.0375 \text{ mequiv/mL} = 18.6 \text{ mL} \times N_2$$
$$N_2 = 0.0403 \text{ mequiv/mL}^*$$

***NOTE:** THAT THE FINAL NORMALITY WAS ROUNDED OFF TO THE SAME NUMBER OF DECIMAL PLACES AND SIGNIFICANT FIGURES AS THE KNOWN NORMALITY OF THE POTASSIUM BI-IODATE.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure (continued)

- Next, divide the sodium thiosulfate normality by the bi-iodate normality to determine the factor as follows: 0.0403/0.0375 = 1.07.

- This factor (1.07) will be multiplied by all Winkler DO titrations to determine the DO in each sample that is titrated.

11. Complete the normality procedure on a duplicate sample, which should agree within ± 0.05 mL of the first test.

12. Keep a written record of this procedure.

Attachment 7.

Temperature – Dissolved Oxygen Calibration Values Table (5340ft altitude)

YSI Dissolved Oxygen Meter Calibration Values (5340 ft. altitude)			
Temperature °C	Setting mg/L O_2	Temperature °C	Setting mg/L O_2
15.0	8.28	21.0	7.33
16.0	8.11	21.2	7.30
17.0	7.94	21.4	7.27
18.0	7.78	21.6	7.24
18.2	7.75	21.8	7.21
18.4	7.72	22.0	7.18
18.6	7.68	22.2	7.15
18.8	7.65	22.4	7.13
19.0	7.62	22.6	7.10
19.2	7.59	22.8	7.08
19.4	7.56	23.0	7.05
19.6	7.53	24.0	6.92
19.8	7.50	25.0	6.79
20.0	7.47	26.0	6.70
20.2	7.44	27.0	6.55
20.4	7.41	28.0	6.43
20.6	7.39	29.0	6.32
20.8	7.36	30.0	6.21

Attachment 8.

Calibration Values for Various Atmospheric Pressures and Altitudes

Calibration Values for Various Atmospheric Pressures and Altitudes					
Pressure			Altitude		Calibration
Inches Hg	mm Hg	Millibars	Feet	Meters	Value %
30.23	768	1023	-276	-84	101
29.92	760	1013	0	0	100
29.61	752	1003	578	85	99
29.33	745	993	558	170	98
29.02	737	983	841	256	97
28.74	730	973	1126	343	96
28.43	722	963	1413	431	95
28.11	714	952	1703	519	94
27.83	707	942	1995	608	93
27.52	699	932	2290	698	92
27.24	692	922	2587	789	91
26.93	684	912	2887	880	90
26.61	676	902	3190	972	89
26.34	669	892	3496	1066	88
26.02	661	882	3804	1160	87
25.75	654	871	4115	1254	86
25.43	646	861	4430	1350	85
25.12	638	851	4747	1447	84
24.84	631	841	5067	1544	83
24.53	623	831	5391	1643	82
24.25	616	821	5717	1743	81
23.94	608	811	6047	1843	80
23.62	600	800	6381	1945	79
23.35	593	790	6717	2047	78
23.03	585	780	7058	2151	77
22.76	578	770	7401	2256	76
22.44	570	760	7749	2362	75
22.13	562	750	8100	2469	74
21.85	555	740	8455	2577	73
21.54	547	730	8815	2687	72
21.26	540	719	9178	2797	71
20.94	532	709	9545	2909	70
20.63	524	699	9917	3023	69
20.35	517	689	10293	3137	68

Attachment 9.

YSI Model 5100 Barometer Calibration Instructions

The YSI Model 5100 has an internal barometer for pressure compensation during AUTO Dissolved Oxygen Calibration. This barometer only needs to be calibrated when it is no longer reading the correct barometric pressure. If the 5100 is kept at a fairly constant ambient temperature (±10°C), the barometer calibration should be accurate for approximately 30 days. As a result the barometer calibration **MUST** be performed monthly

The Model 5000 does not contain a barometer; therefore, the current barometric pressure must be entered before an AUTO Cal is performed. The pressure value displayed is the setting that was entered and stored during the previous calibrations.

From the calibration menu press the [DO CAL] soft-key, then press [NEXT] soft-key until the barometric pressure is flashing and “Barometer” appears in the top right corner of the display.

Using the [UP], [DOWN], and [DIGIT] soft-keys, enter the true local barometric pressure. This corresponds to a reading from a mercury barometer. Do **NOT** use the pressure reported by the weather bureau. Weather bureaus correct pressures to sea level.

NOTE: You may estimate the standard pressure at your altitude by using Attachment 9.

Press [ENTER} to confirm. The message “PRESSURE CALIBRATION SAVED” will be displayed, on the model 5100. The model 5000 will display “PRESSURE SETTING SAVED”, since it does not contain an internal barometer.

NOTE: If you wish to abort before pressing [ENTER}, you may press [MODE] to return to the calibrate menu without saving the new value for barometric pressure. You may also press [NEXT] to select a different parameter (any change made will not be saved).

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Electronic Copy Only

Title: Anions by Ion Chromatography

[EPA 300.0, SW 9056 and 9056A]

Approvals (Signature/Date):

Dave Elkin 12/21/10 Date Wet Chemistry Supervisor	Adam Alban 21 Dec 10 Date Health & Safety Manager / Coordinator
John Morris 12/21/10 Date Quality Assurance Manager	Robert C. Hanisch 12/21/10 Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure describes the determination of the anions fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate, and sulfate in water samples by ion chromatography, based on EPA Method 300.0 and SW-846 Methods 9056 and 9056A.

1.1.2 This procedure can also be applied to leachates from soil samples. The soil leaching procedure is described in SOP DV-WC-0036.

1.1.3 The anions included in this procedure and their routine working ranges for interference-free samples are as follows:

Analyte	CAS Number	Working Range (mg/L)	Working Range (mg/kg)
Fluoride	66-30-0	1.0 – 10	10 – 100
Chloride	1-00-3	3.0 – 50	30 – 500
Nitrite as N	15-90-0	0.5 – 10	5 – 100
Bromide	28-20-0	0.2 – 10	2 – 100
Nitrate as N	25-90-0	0.5 – 10	5 – 100
Phosphate as P	226750-80-0	0.5 – 10	5 – 100
Sulfate	3-03-5	5.0 – 50	50 – 500

Note: The working range can be extended by dilution of the sample.

1.1.4 The reporting limits for the following analytes are based on a 25 µL injection volume:

Analyte	Water RL (mg/L)	Soil RL (mg/kg)
Fluoride	0.5	5
Chloride	3.0	30
Nitrite	0.5	5
Bromide	0.2	2
Nitrate	0.5	5
Phosphate	0.5	5
Sulfate	5.0	50

NOTE 1: Report nitrite (NO_2^-) as N, nitrate (NO_3^-) as N, and phosphate (PO_4^{-3}) as P.

NOTE 2: Depending client or project requirements, reporting limits may be higher than those shown above.

NOTE 3: Reporting limits for soils are based on the DI Leach procedure using a soil to water ratio of 1:10. Client-specific soil to water ratios may differ.

2.0 Summary of Method

2.1 Aqueous samples are measured directly, unless visible particulate is present or the measured concentration exceeds the calibration. Samples with particulate must be filtered before they are injected into the ion chromatograph. High concentration samples must be diluted for analysis. Soil samples are leached using deionized water in accordance with SOP DV-WC-0036, and the water leach is analyzed.

2.2 A small volume of sample is introduced into the ion chromatograph to flush and fill a constant volume loop. The sample is injected into a stream of carbonate-bicarbonate or hydroxide eluent.

2.3 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids.

2.4 The separated anions in their acid forms are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3.0 Definitions

3.1 This procedure includes the drinking water QC terminology from Method 300.0 and the solid waste terminology from SW-846 Methods 9056 and 9056A. Where there are two terms for the same concept, the cross reference is explained below. The frequency and evaluation of these QC samples are discussed in Sections 9 and 10.

3.2 **Calibration Blank (CB)** - A volume of reagent water fortified with the same matrix as the calibration standards, but without the addition of any of the analytes of interest.

3.3 **Laboratory Reagent Blank (LRB, also referred to as a Method Blank)** - For water samples, which do not require any preparation steps, the calibration blank and the method blank are the same thing. When soils are being analyzed, the method blank consists of the same reagents and preparation steps as applied to samples.

3.4 **Field Duplicates (FD)** - Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures. The laboratory project managers are responsible for informing clients of the relevant regulatory requirements for field duplicates.

3.5 **Laboratory Fortified Blank (LFB, also referred to as a Laboratory Control Sample, LCS)** - An aliquot of reagent water or other blank matrix to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a sample and its purpose is to determine whether the methodology is

in control and whether the laboratory is capable of measurements the meet data quality objectives for accuracy and precision.

3.6 **Laboratory Fortified Sample Matrix (LFM, also referred to as a Matrix Spike, MS)** - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix affects the accuracy of the analytical result. The background concentrations of the analyte in the sample matrix must be determined if method analytes or other interference is present in the laboratory environment, the reagent, or the apparatus.

3.7 **Instrument Performance Check Solution (IPC, also referred to as Continuing Calibration Verification Standards, CCV)** - The CCV serves to monitor instrument drift from the beginning to the end of a given analytical sequence.

3.8 **Linear Concentration Range (LCR)** - The concentration range over which the instrument response is linear.

3.9 **Quality Control Sample (QCS)** - The QCS provides an independent verification of the accuracy of calibration standards and instrument performance. For the purposes of this SOP, the second-source ICV provides this verification.

3.10 Other quality control terms (e.g., MDL and PE sample) are defined in the Glossary Section of the Quality Assurance Manual.

4.0 Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent to 100 mL of each standard and sample. However, for routine samples, this problem is not severe enough to require this procedure.

4.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate (with carbonate/bicarbonate eluent) and other low molecular weight organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Nitrite	Oxidizer Toxic	None established	**Danger - strong oxidizer. Contact with other material may cause fire.** Toxic by inhalation; causes irritation to the respiratory tract and systemic poisoning, including intense cyanosis, nausea, dizziness, vomiting, collapse, spasms of abdominal pain, rapid heart beat, irregular breathing, coma, convulsions, and death due to circulatory collapse. Causes irritation, redness, and pain in contact with skin. May be absorbed through the skin causing systemic poisoning. Causes irritation, redness, and pain in contact with eyes.
Sodium Nitrate	Oxidizer	None established	**Danger - strong oxidizer. Contact with other material may cause fire.** Inhalation of dust irritates respiratory tract; symptoms may include coughing and shortness of breath. May cause irritation, redness, itching, and pain in contact with skin and eyes.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Fluoride	Poison	2.5 mg/m3 (TWA as F)	**Highly Toxic!** Inhaled or swallowed, this compound can cause fluoride poisoning. Early symptoms include nausea, vomiting, diarrhea, and weakness. Later effects include central nervous system effects, cardiovascular effects, and death. Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation in contact with skin, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
1 – Always add water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- **Balance:** Analytical, capable of accurately weighing to the nearest 0.0001 g. Checked for accuracy each day of use in accordance with DV-QA-0014.
- Conductivity meter
- **Ion Chromatograph:** Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases, and detectors.
- **Anion guard column:** A protector of the separator column. If omitted from the system, the retention times will be shorter. Usually packed with a substrate that is the same as on the separator column. Dionex AG14, 4 x 50 mm, Dionex AG17-C, or equivalent.
- **Anion separator column:** The separation shown in Figure 1 was generated using a Dionex AS14 column. An alternate column, e.g., Dionex AS17-C, may be used if comparable resolution of the peaks is obtained, and all QC requirements can be met.
- **Anion suppressor device:** Dionex Anion Self-Regenerating Suppressor, or equivalent.
- **Detector:** Conductivity cell, approximately 1.25 µL internal volume, Dionex, or equivalent.
- Dionex ion chromatography software.
- Interfaced computer with printer.
- **Computer Software and Hardware:** Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

6.2 Supplies

- Volumetric Flasks (Class A): various sizes
- Eppendorf Pipettes, varying volumes
- Autosampler vials and caps
- 0.2 micron nylon syringe filters, and other miscellaneous filtration materials
- Disposable plastic beakers
- Other miscellaneous laboratory supplies

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all materials listed below must be reagent grade, unless listed otherwise.

7.1 Reagent Water

Deionized water, Milli-Q grade, ≥ 18 Megohm-cm.

7.2 Eluent for New Generation ICs with column AS17-C

New chromatographs use the Dionex RFIC™ systems, which employs an eluent generator. The eluent generators are purchased from Dionex. The eluent generators use electrolysis to generate a potassium hydroxide eluent, the concentration of which is controlled by the instrument software.

7.3 Eluent Concentrate:

The eluent concentrate for IC 3 (column AS14) is prepared at concentrations of the eluent species that are 100 times greater than used in the eluent solution. Dissolve 37.12 g of sodium carbonate (Na_2CO_3) and 8.4 g of sodium bicarbonate ($NaHCO_3$) in reagent water and dilute to 1 liter. This solution may be purchased commercially.

NOTE: A small amount of eluent concentrate, i.e., 50 µL in 5 mL of sample, is added to each sample, QC sample, and standard injected to eliminate the water dip. The eluent concentrate is not added to samples and standards analyzed on newer generation ion chromatographs that employ an eluent generator.

7.4 Eluent Solution:

Dilute 20 mL of the eluent concentrate (7.3) in reagent water to 2 liters.

NOTE: The bicarbonate/carbonate eluent solution is used in older generation ion chromatographs. New chromatographs use the Dionex RFIC™ systems, which employs an eluent generator. The system uses electrolysis to generate a potassium hydroxide eluent, the concentration of which is controlled by the instrument software.

7.5 Stock Solutions (1,000 mg/L)

All stock solutions are purchased from commercial sources.

WARNING! Sodium fluoride is highly toxic. Exercise extreme caution when working with sodium fluoride. See Section 5.2.

7.5.1 Fluoride Stock Solution (1.00 mg of F^- in 1.00 mL of solution)

In a 1-liter volumetric flask, dissolve 2.21 g of sodium fluoride (NaF) in reagent water, and dilute to volume with reagent water. Store in chemical-resistant glass or polyethylene.

7.5.2 Chloride Stock Solution (1.00 mg of Cl^- in 1.00 mL of solution)
Dry sodium chloride (NaCl) for 1 hour at 600 °C, and cool in a desiccator. In a 1-liter volumetric flask, dissolve 1.6484 g (weigh to the nearest mg), of the dry salt in reagent water and dilute to volume with reagent water.

7.5.3 Nitrite Stock Solution (1.00 mg of NO_2^- as N in 1.00 mL of solution)
Place approximately 10.0 g of sodium nitrite ($NaNO_2$) in a 125 mL beaker and dry to a constant weight (about 24 hours) in a desiccator. In a 1-liter volumetric flask, dissolve 4.9257 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly.

- Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used each day.
- Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170 °C in an air oven.

7.5.4 Bromide Stock Solution (1.00 mg Br^- in 1.00 mL of solution)
Dry approximately 5.0 g of sodium bromide (NaBr) for 6 hours at 150 °C, and cool in a desiccator. In a 1-liter volumetric flask, dissolve 1.2877 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

7.5.5 Nitrate Stock Solution (1.00 mg NO_3^- as N in 1.00 mL of solution)
Dry approximately 10.00 g of sodium nitrate (NaNO3) at 105 °C for 24 hours. In a 1-liter volumetric flask, dissolve 6.0679 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

7.5.6 Phosphate Stock Solution (1.00 mg PO_4^{-3} as P in 1.00 mL of solution)
Dry approximately 10.0 g of potassium dihydrogen phosphate (KH2PO4) for 1 hour at 105 °C and cool in a desiccator. In a 1-liter volumetric flask, dissolve 4.3937 g (weigh to the nearest mg) of the dry salt in reagent water and dilute to volume with reagent water.

7.5.7 Sulfate Stock Solution (1.00 mg SO_4^{-2} in 1.00 mL of solution)
Dry approximately 5.00 g of potassium sulfate (K2SO4) at 105 °C for 1 hour and cool in a desiccator. In a 1-liter volume flask, dissolve 1.8141 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

NOTE: The stability of the stock standards is at least 1 month when stored at 4 °C. Dilute working standards should be prepared weekly, except those that contain nitrite and phosphate should be prepared fresh daily.

7.6 Second Source Stock Solutions (1,000 mg/L)
Stock solutions for each anion of interest are purchased at the same concentration but from a vendor other than the one that supplied the Stock Solutions described in Section 7.5. The second source stock solutions are used to prepare the ICV Intermediate Stock Solution (Section 7.8).

7.7 Calibration (CAL) Intermediate Stock Solution

Prepare the intermediate stock solution by combining the prescribed aliquots of each of the stock solutions described in Section 7.5(see table below) in a 100 mL volumetric flask. Dilute to volume with reagent water. Include all anions of interest, but omit all others.

Analyte	mL of Stock	Final Volume (mL)	Final Concentration (mg/L)
Fluoride	5.0	100	50
Chloride	25.0	100	250
Nitrite as N	5.0	100	50
Bromide	5.0	100	50
Nitrate as N	5.0	100	50
Phosphate as P	5.0	100	50
Sulfate	25.0	100	250

NOTE: The aliquots and final concentrations shown above are only recommended and assume a 1000 mg/L stock solution. Other aliquots and final concentrations may be used.

7.8 ICV Intermediate Stock Solution

Prepare the intermediate ICV stock solution by combining the prescribed aliquots of the second source (ICV) stock solutions (Section 7.6) in a 10 mL volumetric flask. Dilute to volume with reagent water. Include all anions of interest, but omit all others. The following table prescribes aliquot sizes for the individual anion stock solutions.

Analyte	mL of Stock	Final Volume (mL)	Final Concentration (mg/L)
Fluoride	0.5	10	50
Chloride	2.5	10	250
Nitrite as N	0.5	10	50
Bromide	0.5	10	50
Nitrate as N	0.5	10	50
Phosphate as P	0.5	10	50
Sulfate	2.5	10	250

NOTE: The aliquots and final concentrations shown above are only recommended and assume a 1000 mg/L stock solution. Other aliquots and final concentrations may be used.

7.9 Working Standards

Prepare working standards by pipetting 10mL aliquots of the CAL Intermediate Stock Solution (Section 7.7) into 100 mL volumetric flasks. Dilute to volume with reagent water. Refer to the following tables for the concentration of each analyte in the calibration standard.

7.9.1 Calibration Standard #1

Dilute 10 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.1
Chloride	0.5
Nitrite as N	0.1
Bromide	0.1
Nitrate as N	0.1
Phosphate as P	0.1
Sulfate	0.5

7.9.2 Calibration Standard #2

Dilute 50 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.5
Chloride	2.5
Nitrite as N	0.5
Bromide	0.5
Nitrate as N	0.5
Phosphate as P	0.5
Sulfate	2.5

7.9.3 Calibration Standard #3

Dilute 100 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	1.0
Chloride	5.0
Nitrite as N	1.0
Bromide	1.0
Nitrate as N	1.0
Phosphate as P	1.0
Sulfate	5.0

7.9.4 Calibration Standard #4

Dilute 0.4 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	4.0
Chloride	20.0
Nitrite as N	4.0
Bromide	4.0
Nitrate as N	4.0
Phosphate as P	4.0
Sulfate	20.0

7.9.5 Calibration Standard #5

Dilute 0.8 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	8.0
Chloride	40.0
Nitrite as N	8.0
Bromide	40.0
Nitrate as N	8.0
Phosphate as P	8.0
Sulfate	40.0

7.9.6 Calibration Standard #6

Dilute 1 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	10.0
Chloride	50.0
Nitrite as N	10.0
Bromide	10.0
Nitrate as N	10.0
Phosphate as P	10.0
Sulfate	50.0

NOTE: The aliquots and final concentrations shown above are recommended for calibration. Other aliquots and final concentrations may be used.

7.9.7 Lower Limit of Quantitation (LLOQ)
Dilute 20 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.2
Chloride	1.0
Nitrite as N	0.2
Bromide	0.2
Nitrate as N	0.2
Phosphate as P	0.2
Sulfate	1.0

7.10 Initial Calibration Verification (ICV)
Dilute 1.0 mL of the second source ICV Intermediate Stock Solution (Section 7.8) to 25 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	2.0
Chloride	10.0
Nitrite as N	2.0
Bromide	2.0
Nitrate as N	2.0
Phosphate as P	2.0
Sulfate	10.0

7.11 Continuing Calibration Verification (CCV) and Laboratory Control Sample (LCS) Solution
Prepare the CCV and LCS spike solution by diluting 10.0 mL of the ICAL Intermediate Stock Solution (Section 7.7) to 100 mL with reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	5.0
Chloride	25.0
Nitrite as N	5.0
Bromide	5.0
Nitrate as N	5.0
Phosphate as P	5.0
Sulfate	25.0

NOTE: The aliquots and final concentrations shown above are recommended. Other aliquots and final concentrations may be used.

7.12 Stock Spiking Solutions (5000 mg/L chloride, sulfate and 1000 mg/L fluoride, bromide, nitrate, phosphate/1000 mg/L nitrite)

For the spiking solution, solids are dried and desiccated as listed in Section 7.4 for each individual component of the solution. Two solutions are made as summarized in the following two tables. Alternatively, a commercially prepared solution may be used.

Stock Spiking Solution #1

Analyte	Mass of Solid (g)	Final Volume (mL)	Anion Conc. (mg/L)
Sodium Fluoride	2.21	1000	1000
Sodium Chloride	8.2424	1000	5000
Sodium Bromide	1.2876	1000	1000
Sodium Nitrate	6.068	1000	1000
Potassium Dihydrogen Phosphate	4.3936	1000	1000
Potassium Sulfate	9.0704	1000	5000

Stock Spiking Solution #2

Analyte	Mass of Solid (g)	Final Volume (mL)	Anion Conc. (mg/L)
Sodium Nitrite	4.9256	1000	1000

7.13 Spikes for the Matrix Spike and Matrix Spike Duplicate (MS/MSD)

A working solution consisting of 10 mL of Stock Spiking Solution #1 and 10 mL of Stock Spiking Solution #2 is used. The MS is prepared by adding 50 µL of this working solution to a 5 mL aliquot of the selected sample. The MSD is prepared by adding 50 µL of this working solution to a second 5 mL aliquot of the same sample. This will result in the following anion concentrations ("true values") in the spiked sample: 25 mg/L for chloride and sulfate, and 5 mg/L for fluoride, nitrite, bromide, nitrate, and phosphate.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Analyte	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Fluoride	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Chloride	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Nitrite as N	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Bromide	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Nitrate as N	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Phosphate as P	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Sulfate	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3

NOTE: Soil leachates follow the same preservation and holding times as the water samples, starting from the time of extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. The process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program. Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.3 Attachment 1 reconciles the various QC requirements specified in the reference methods with the QC requirements specified in this SOP.

9.4 Before analyzing samples, the laboratory must establish a method detection limit (MDL) as described in Section 12.1 and the linear concentration range (LCR) as described in Section 9.7.6. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details.

9.5 Batch Definition
Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.6 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.6.1 Method Blank (same as Laboratory Reagent Blank, LRB)
A method blank (MB) is required with every batch of 20 or less samples. The MB is deionized water taken through the procedure as if it were a sample.

Acceptance Criteria: The MB must not contain anions of interest above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, laboratory contamination is suspected and corrective action must be taken before continuing. All samples associated with the failed blank must be reanalyzed

9.6.2 Laboratory Control Sample (same as Laboratory Fortified Blank, LFB)

One Laboratory Control Sample (LCS) is required with each analytical batch. Depending on client or project requirements, an LCS duplicate may also be analyzed. The LCS and LCSD are prepared as described in Section 7.11. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch.

Acceptance Criteria: For Method 300.0, the LCS recovery for each analyte of interest must be within 90-110%. For Method 9056, the LCS recovery for each analyte of interest must be within statistical control limits, not to exceed 85-115%. For Method 9056A, the LCS recovery for each analyte of interest must be within statistical control limits, not to exceed 80-120%.The absolute value of the relative percent difference (RPD) between the LCS and LCSD must be ≤10%. Statistical control limits are set at ± 3 standard deviations around the historical mean. The process of establishing control limits is described in more detail in Policy DV-QA-003P. Control limits are maintained in the LIMS.

Corrective Action: If the LCS recovery falls outside of the established control limits, and/or when the RPD for the LCS/LCSD exceeds the RPD limit, then check instrument conditions and the standards being used for problems. Correct any problems before continuing. Reanalyze all samples associated with the failed LCS.

9.6.3 Matrix Spike / Matrix Spike Duplicate (MS/MSD, same as Laboratory Fortified Matrix)

For Method 9056, one MS/MSD pair is required with each analytical batch of 20 or fewer samples. For Method 300.0, one MS is required for every 10 routine samples. Also note that some programs (e.g., North Carolina and South Carolina) require an MS/MSD pair for every 10 samples. The MS and MSD are prepared as described in Section 7.13.

Acceptance Criteria: The recovery of each anion of interest must be within the established statistical control limits. Statistical control limits are set at ± 3 standard deviations around the historical mean, and must be within 80-120%. The relative percent difference (RPD) between the MS and MSD must be less than 20%, or less than the established control limit, depending on project requirements. The process of establishing control limits is described in more detail in Policy DV-QA-003P. Control limits are maintained in the LIMS.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS

and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly;
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses); and
- Document the failure in an NCM and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and document the failure in an NCM.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.7 **Instrument QC** - The following quality control samples are prepared with each analytical instrument run.

9.7.1 Initial Calibration Verification (ICV)

The second-source ICV, as described in Section 7.10, is analyzed immediately following the ICAL.

Acceptance Criteria: The ICV recovery for each anion must be 90-110%. The retention time for each analyte in the ICV must be within ± 10% of the established retention time for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, repeat the test. If the test fails on the second attempt, then the problem must be investigated and the instrument recalibrated for the failed analyte(s).

9.7.2 Initial Calibration Blank (ICB)

An ICB is analyzed following the ICV.

Acceptance Criteria: The result must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument

maintenance. The instrument must be recalibrated.

9.7.3 Continuing Calibration Verification (CCV, same as IPC in Method 300.0)

A CCV is required after every 10 or fewer samples and after the last sample.

Acceptance Criteria: The CCV recovery must be 90-110%. The retention time for each analyte in the CCV must be within ± 10% of the established retention time for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, the instrument must be recalibrated, and all samples analyzed since the last successful CCV must be reanalyzed.

9.7.4 Continuing Calibration Blank (CCB)

A CCB is analyzed after each CCV.

Acceptance Criteria: The result must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. The instrument must be recalibrated, and all samples analyzed since the last successful CCB must be reanalyzed.

9.7.5 Lower Limit of Quantitation (LLOQ)

A LLOQ is required for method 9056A to verify the data reporting limit for each analyte.

Acceptance Criteria: The LLOQ recovery must be ± 50% of the true value.

Corrective Action: If the recovery is outside of the acceptance limits, the instrument must be recalibrated.

9.7.6 Linear Concentration Range (LCR)

9.7.6.1 The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or a significant change (eg. Change to column type, eluent or instrument pressures) in instrument configuration is made.

9.7.6.2 The initial demonstration of linearity must use a sufficient number of standards to ensure that the resulting curve is linear.

9.7.6.3 The semi-annual verification of linearity must use a minimum of a blank and three standards. If the recovery for any analyte falls outside of 90-110%, linearity must be reestablished.

9.7.6.4 Linearity study data are maintained by the Wet Chemistry group leader.

9.7.7 Retention Time Study

The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms. See Section 10.3.4 for detailed instructions on performing the retention time study.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Screen the conductivity of samples prior to analysis to determine whether the samples require dilution using a conductivity meter. Record the conductivity on a sample observation benchsheet if the conductivity is > 3000 µmhos/cm.

10.1.2 If the conductivity is > 3000 µmhos/cm, dilute as necessary with reagent water, based on the following calculation, rounding up to the smallest round

$$\text{dilution factor} = \frac{conductivity}{3000}$$

dilution that will bring the conductivity of the dluted sample to under 3000.

NOTE: If a sample requires filtration, prior to being loaded on the instrument, the method blank **must** also be filtered.

10.2 Calibration

10.2.1 An initial calibration is performed every three months, or as needed, based on instrument performance and maintenance.

10.2.2 Calibrate the instrument at six levels. See Section 7.9 for preparation of calibration standards.

NOTE: It is generally NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason is clearly documented, for example a cracked tube, and a minimum of five levels remain.

10.2.3 Construct a calibration curve using a weighted linear regression. Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

Acceptance Criteria: The absolute value of the correlation coefficient must be 0.995 or greater.

Corrective Action: If the correlation coefficient is less than the acceptance limit, recheck instrument conditions and calibration standards. Samples cannot be analyzed until the initial calibration is successful.

10.2.4 Attachment 2 summarizes the recommended operating conditions for the ion chromatograph.

10.2.5 Attachment 3 is an example chromatogram. Included in this chromatogram are example retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the data quality objectives can be met.

10.2.6 Check system calibration daily by analyzing an ICV and ICB (see Sections 9.7.1 and 9.7.2) and, if required, recalibrate.

10.3 Sample Analysis

10.3.1 When using the older generation ion chromatographs that do not employ eluent generation, add 50 µL of eluent concentrate (Section 7.3) to 5 mL of each standard, sample, and QC sample prior to injecting.

10.3.2 Load samples into the autosampler according to the schedule. The instrument will flush and load the sample loop for injection. See Attachment 2 for sample loop specifications. The instrument software detects and integrates peaks in the resulting chromatograph.

10.3.3 Following is a typical analytical sequence:

ICAL
ICV and ICB
LCS and LCSD (if LCSD required)
Method Blank
10 injections
CCV and CCB
10 injections
CCV and CCB
10 injections
CCV and CCB

10.3.4 Retention Times and Anion Identification

10.3.4.1 The width of the retention time window used to make identifications is based on measurements of actual retention time variations of standards over 72 hours. (This should be done prior to analysis of samples since the retention time window must be entered into the software before starting the run). The retention time windows should be re-evaluated and a retention time study run when significant changes (eg. Change to column type, eluent or instrument pressures) are made to the instrument.

10.3.4.2 Record the retention time of each calibration standard on at least 3 different days. Calculate the standard deviation for each analyte. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.3.4.3 The calibration curve is verified each working day or whenever the eluent is changed by analyzing an ICV and ICB, and after every 10 injections by analyzing a CCV and CCB. If the retention time for any analyte varies from the expected values (±5% for phosphate and sulfate and ± 10% for fluoride, chloride, nitrite, bromide and nitrate) a new calibration curve must be prepared.

10.3.4.4 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze. The addition of one to two times the sample concentration normally provides the best peak height for analyte identification.

NOTE: Concentration can affect retention time and cause peak migration. Late eluting species, e.g., nitrate and sulfate, exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration may produce poor resolution or misidentification. If a peak has shifted outside of its retention time window (as confirmed by a CCV or Matrix Spike), change the window in the software and reprocess the chromatogram. Document the reason for reprocessing the chromatogram along with the date and initials.

10.3.4.5 Should more complete resolution be needed between peaks, the eluent can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

10.3.5 If the response for the peak exceeds the working range of the system, dilute the sample with reagent water and reanalyze.

11.0 Calculations / Data Reduction

11.1 Using the computer and the Dionex software package, prepare a linear calibration curve for each analyte by plotting instrument response against standard concentration. The software calculates a calibration function in the following form:

$$Y_i = SX_i + I$$

Where:

Y_i = Instrument response (peak area) for specific anion in the i^{th} calibration standard.
X_i = Concentration of anion in the i^{th} calibration standard, mg/L.
S = Slope of calibration curve determined by linear regression analysis.
I = Intercept of calibration curve determined by linear regression analysis.

11.2 The anion concentration in the injected sample is calculated by solving the calibration function for Xi as follows:

$$X_i = \frac{Y_i - I}{S}$$

Where:

X_i = Calculated concentration of the i^{th} sample at the instrument, mg/L.
Y_i = Instrument response for the i^{th} sample (peak area).
I = Intercept of the calibration curve.
S = Slope of the calibration curve.

11.3 If the sample was diluted, the final anion concentration in the original sample is calculated as follows:

$$X_f = X_i \times DF$$

Where:

X_f = Anion concentration in original sample, mg/L.
X_i = Calculated concentration of sample at the instrument, mg/L.
DF = Dilution factor.

11.4 For soil leachates, the concentration in the original soil sample is calculated as follows:

$$X_s = X_i \times DF \times \frac{V}{M_s}$$

Where:

X_s = Anion concentration in original soil sample, mg/kg.
X_i = Calculated concentration of sample at instrument, mg/L.
DF = Dilution factor, if applicable.
V = Volume of leachate, L.
M_s = Mass of original soil sample, kg.

11.5 Report only those values that are less than the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

11.6 Use the appropriate data qualifier (i.e., "flag") to indicate when a sample requires dilution due to high conductivity. In most cases, a "G" flag denotes dilution due to matrix effects, and a "Q" indicates dilution to bring the sample into the calibration range. Special flagging conventions may apply depending on client or project requirements.

11.7 All results are subject to two levels of review, which are documented on the form shown in Attachment 4.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

NOTE: EPA method 300.0 requires an MDL study to be performed every 6 months.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

- Initially the analyst must perform an MDL study (see section 12.1).
- Four aliquots of the QC check sample (independent source from the calibration) 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 standard.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

- 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.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive ICVs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

The use of ion chromatography eliminates the need to use a variety of hazardous reagents required by the other approved methods for the determination of the same anions.

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- IC process waste from older generation ion chromatographs – aqueous carbonate/bicarbonate eluent waste: Non-hazardous
- IC process waste from new generation ion chromatographs with hydroxide eluent generating system: Excess Sample – Aqueous – Waste Stream W

- Expired aqueous reagents/standards – Contact Waste Coordinator
- Expired solid chemicals – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW846, 3rd Edition, Final Update IIIB (December 1996), Method 9056, "Determination of Inorganic Anions by Ion Chromatography", Revision 0, September 1994.

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW846, 4th Edition, Final Update IV, Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", Revision 1, February 2007.

15.3 Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 300.0	Method 300.0 includes a requirement for a second-source quality control standard (QCS), which is to be run quarterly and be within ±10% of the expected value. This SOP establishes calibration accuracy every day of operation using a second-source initial calibration verification (ICV) standard.
2	EPA 300.0	Method 300.0 specifies that target analytes must be less than the MDL in the Laboratory Reagent Blank. TestAmerica Denver QA policy (Policy DV-QA-003P) defines the acceptance limit for the method blank as the laboratory reporting limit (RL) and not the MDL. If specified in client or project requirements, the method blank acceptance limit may be set at the MDL.
3	EPA 300.0, SW 9056 and SW 9056A	Methods 9056 and 9056A specifies bomb combustion for solid waste samples. Method 300.0 specifies water leaching for solid samples. This SOP specifies water leaching and references SOP DV-WC-0036 for the deionized water leach procedure. In this respect, this SOP complies with Method 300.0, but deviates from Method 9056A.

17.0 Attachments

Attachment 1: Quality Control Summary
Attachment 2: Suggested Standard Instrument Operating Parameters
Attachment 3: Example Ion Chromatogram
Attachment 4: Example Data Review Checklist

18.0 Revision History

- Revision 7.2, dated 23 December 2010
  - Annual Technical Review

- Revision 7.1, dated 04 December 2009
  - Added bullet in section 6.1 concerning the Master List of Documents.
  - Added note on filtration to section 10.1.
  - Updated Attachment 4

- Revision 7, dated 19 June 2009
  - Added Lower limit of quantitation information.
  - Updated reagent and standard preparation information.
  - Made minor formatting and grammatical corrections.
  - Added 6 month MDL requirement note to section 12.1
  - Updated Attachment 4.

Revision 6, dated 22 February 2008

- Update the recipes for the eluent concentrate and eluent solution to current practices.
- Added the information concerning the eluent generators and need to purchase them from Dionex.

- Revision 5, dated 22 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Moved the reporting limit table to Section 1.
  - Updated attachments with the TestAmerica logo.

- Revision 4, dated 24 July 2007
  - The method Reference has been changed for SW846 from Method 9056 to method 9056A.
  - The company name has been changed from STL to TestAmerica.

- Revision 3, dated 28 February 2006
  - Revised Sections 5, 14, and 15 (Safety, Pollution Prevention, and Waste Management, respectively) to comply with STL Corporate requirements.
  - Updated formatting to comply with current STL Denver guidance (Policy QA-001).
  - Updated the reporting limit table in Section 1.1.4 to reflect current reporting limits.
  - Expanded discussion of the matrix spike quality control criteria (Section 9.6.3) to be consistent with Policy QA-003, Quality Control Program.
  - Removed details concerning Method Performance, and included references to the applicable STL Denver SOPs.
  - Expanded Attachment 1, Quality Control Summary, to include references to the source methods.

- Revision 2, dated 6 September 2002
  - Calibration standards updated and includes addition of eluent concentrate to

working standards.
- Eluent concentrate recipe added.
- Changed reporting limit tables to reflect current reporting limits.
- Added requirement for MS/SD pair per 10 samples for certain clients or agencies.
- The spike concentration was increased to avoid overly diluting the unspiked sample when matrix spikes are prepared.
- The Method Performance Section 13 was expanded.
- A Data Review Checklist was added to the end of the SOP.

- Revision 1, dated 9 June 1999
  - The size of the injection loop for low level analysis is changed from 300 uL to 25 uL, which is the manufacturer's recommendation for the latest equipment.

Attachment 1.

Quality Control Summary

QC Samples	Frequency	Acceptance Criteria	Corrective Action	Reference Method Equivalent
Initial Calibration Verification (ICV)	Immediately following the initial calibration, and at the start of each day prior to sample analysis, or whenever the eluent is changed.	90 - 110% of true value RT must be ± 10% of established RT	Repeat once, and recalibrate and reanalyze if it fails a second time.	QC Reference Sample (9056A) IPC and QCS (300.0)
Initial Calibration Blank (ICB)	After the ICV and prior to sample analysis.	≤ the Reporting Limit	Re-prepare and reanalyze	Calibration Blank (300.0)
Laboratory Control Sample (LCS)	1 per QC batch	Within laboratory historical limits	Recalibrate and reanalyze all samples associated with unacceptable LCS	LFB (300.0)
Matrix Spike Sample (MS/MSD)	1 MS/MSD pair per QC batch for 9056. 1 MS/MSD pair per 10 samples for 300.0.	%R within laboratory historical limits and RPD ≤ laboratory historical limits.	If LCS and CCVs are in control, then document in an NCM, unless project requires reanalysis.	Matrix Spike (9056A) LFM (300.0)
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence.	90 - 110% of true value	Repeat. If repeat fails, recalibrate and reanalyze all samples since the last acceptable CCV.	Mid-range Calibration Standard (9056A) IPC (300.0)
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< the Reporting Limit	Repeat. If repeat fails, recalibrate and reanalyze all samples since the last acceptable CCB.	Calibration Blank (300.0)
Method Blank (MB)	1 per QC batch	≤ the Reporting Limit	See Section 9.6.1 or Policy QA-003.	LRB (300.0)

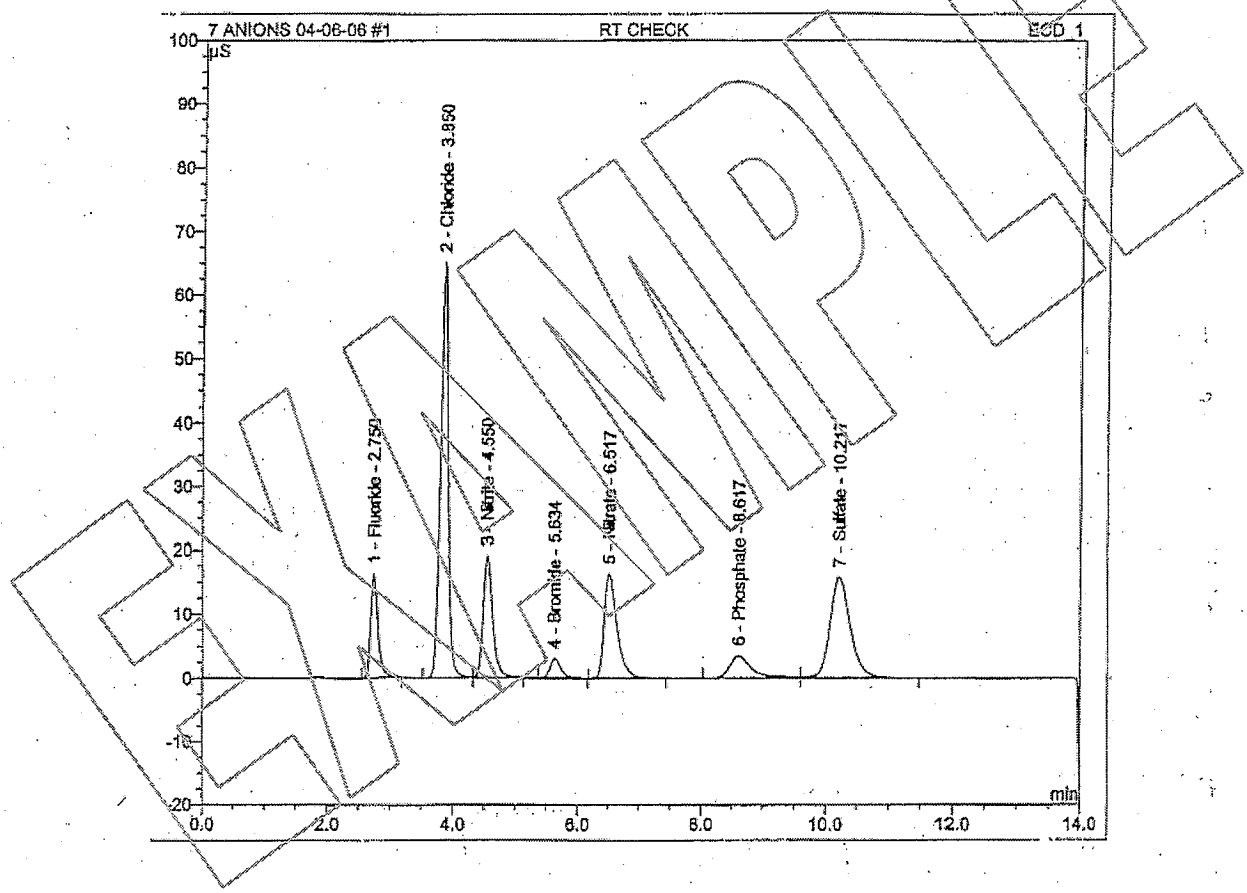
Attachment 2.

Suggested Standard Instrument Operating Procedures

Instrument Conditions	
Anion Guard Column	Dionex AG 4A or AG17-C
Anion Separator Column	Dionex AS4A or AS17
Supressor Device	SRS Ultra II Self-Regenerating Suppressor
Pump Rate	1.2 mL/min or 1.0 mL (depending on specific instrument used)
Sample Loop	10 µL
Eluent	Older Generation Instruments: 1.0 mM sodium bicarbonate, 3.5 mM sodium carbonate Dionex RFIC™ Systems: Potassium hydroxide, generated by electrolysis of water, in the range of 10 to 40 mM
Detector Output	Baseline conductivity should be between 15 - 16 µS prior to sample analysis.

Attachment 3.

Example Ion Chromatogram



Attachment 4.

Example Data Review Checklist

TESTAMERICA Denver

TestAmerica
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Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Input

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lot Dates report checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD/True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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Title: Alkalinity by Automated Titration [EPA 310.1, SW9040B, SM2320B, SM 4500-H+B]

Approvals (Signature/Date):

Dave Elkin — Date 11/2/10
Wet Chemistry Supervisor

Adam Alban — Date 04 Nov 10
Health & Safety Manager / Coordinator

John P. Morris — Date 11/2/10
Quality Assurance Manager

Robert C. Hanisch — Date 11/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure is to be used for the determination of alkalinity. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can also be calculated.

Analyte	CAS Number
Alkalinity	N/A

1.1.2 This method is applicable to all ranges likely to be encountered. As a practical matter, samples with an alkalinity greater than about 1200 mg/L as calcium carbonate require a reduced volume or stronger titrant in order to keep the titrant volume to a reasonable amount.

2.0 Summary of Method

2.1 Samples are analyzed by each method simultaneously on an automated titrator as follows:

2.1.1 The pH is determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.1.2 Alkalinity is determined by titration of the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as calcium carbonate. Samples for alkalinity should not be altered (i.e., filtered or diluted).

3.0 Definitions

3.1 **pH** - At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of the ionic interactions in all but very dilute solutions, it is necessary to use the "activity" of the hydrogen ion and not its molar concentration. The approximate equivalent to molarity can be presumed only in very dilute solutions. A logarithmic scale is used for pH in order to express a wide range of hydrogen ion activities. Neutral pH is 7.0 at 25 °C, while acidic pH's are <7 and basic pHs are >7.

3.2 **Alkalinity** – A measure of the acid-neutralizing capacity of water.

4.0 Interferences

4.1 The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids having a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of

the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a "low sodium error" electrode.

4.2 The pH electrode may exhibit slow or noisy response with high purity waters due to the lack of ionic strength.

4.3 Coatings of oil and particulate matter may impair electrode response.

NOTE: If the electrode becomes coated with oil, immerse in a mild detergent solution, rinse well with deionized water, and recalibrate. If this fails, try rinsing in 10% HCl.

4.4 Temperature variations will change the pH of the samples and also affect electrode response. Electronic temperature correction must be used to correct for electrode response.

4.5 Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and bicarbonate only, the presence of these substances will cause high results.

4.6 The pH 4.5 is the routine endpoint for total alkalinity. This assumes the normal carbon dioxide/bicarbonate/carbonate/hydroxide mass action interrelationships for natural waters. Other types of waters can have other interrelationships that might dictate the use of a different endpoint.

4.7 Samples not in equilibrium with the atmosphere may exhibit changes in pH and in the distribution of the various forms of alkalinity when exposed to the atmosphere. The sample containers should be filled completely and kept closed until just prior to the analysis. The analysis should be performed as soon as possible.

4.8 Particulates in the sample may affect the alkalinity results. This interference can cause an error in the ion balance calculation.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Man-Tech Autotitator, consisting of:
  - Burivar I/2 Buret Module
  - Titrasip Titration Module
  - PC-Tis Interface Module
  - PC running PC-Titrate software
- Radiometer Autotitrator, consisting of:
  - SAC 80 Sample Changer
  - TIT 85 Titrator
  - ABU 80 Autoburette
- Combination pH Electrode – Epoxy-covered glass, with temperature correction, Ross Sure-Flow or equivalent.

6.2 Supplies

- Tubes to fit autosampler. These must be thoroughly rinsed to remove all traces of salt if reused.
- Pipette calibrated to 10.0 mL, and disposable tips.
- Miscellaneous laboratory apparatus and glassware.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software/hardware located on G\QA\Read\Master List of Documents\Master List of Documents and Software/hardware.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 All of the standard materials are obtained from commercial sources, and must be NIST traceable.

7.2 **pH Buffers:** 4, 7, and 10.

7.3 **Alkalinity Standards:**

7.3.1 **Sodium Carbonate solution (1N)**
This standard is purchased commercially.

7.3.2 **Sodium Carbonate solution (200 mg/L as CaCO3)**
Pipette 4mL of the 1N Sodium Carbonate Solution into a 1 liter volumetric flask. Bring to volume with de-ionized water.

7.3.3 **0.02 N Sulfuric Acid (Alkalinity Titrant)**
Purchase from a commercially available source.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Method	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
2320B	HDPE	1 liter	Cool 4 ± 2°C	14 Days	40 CFR Part 136.3
9040B	HDPE	1 liter	None	Analyze Immediately	40 CFR Part 136.3

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit

Quality Controls	Frequency	Control Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	± 10% of the true value
Sample Duplicate - pH	1 in 20 or fewer samples[2]	≤0.05 units
Sample Duplicate - Alkalinity	1 in 20 or fewer samples	≤ 10% RSD

[1] MB is not possible for pH
[2] Some programs (e.g., South Carolina and North Carolina) require duplicates to be analyzed at a 10% frequency.

9.2 Instrument QC

Initial Check and Continuing Calibration Verification

- Calibration verification standard (CCV) and a continuing calibration blank (CCB) are to be analyzed after ten samples and at the end of the run.
- The 200 mg/L standard (7.3.2) is used for the alkalinity Initial check and CCV.
- The pH 7 buffer is used for the pH CCV.
- A blank is not appropriate for pH analysis.

10.0 Procedure

10.1 Sample Preparation

Allow the sample to come to room temperature before analyzing.

10.2 Calibration

10.2.1 Initial Calibration for pH

- The pH meter is calibrated each day of operation.
- Be sure that the reference electrode has been filled with 3M potassium chloride.
- Calibration of the pH meter is done using pH 4,7, and 10 buffers.
    - Fill the first four tubes in the autosamples with the following order of samples: pH 4 buffer, pH 7 buffer, pH 10 buffer, and deionized water.
    - Click on the button "PH CALIBRATION 4-7-10" and follow the screens to calibrate.
    - When calibration has finished, go to titrator and choose "examine calibrations." Print the calibration if it is valid.
    - Open up "documents" and select the bottom option (Alk-new) to update the buffers and standards. Update info and print this out.

10.3 Sample Analysis

10.3.1 Be sure the titrant reservoir of 0.02 N sulfuric acid is at least half full. Be sure that there are no air bubbles in the line. Fill deionized water container used for rinses to the top.

10.3.2 Click on the button "Conductivity – pH – Alkalinity". Click load template and load the appropriate template. Add the sample IDs to the schedule and save the template.

10.3.3 Begin to load the autosampler as indicated by the schedule, using approximately 40 mL in each tube. Click the button "start" when ready to begin analysis.

10.3.4 The titrator has been programmed to deliver a maximum volume of 25mL titrant. Samples requiring more than this should be reanalyzed using a smaller aliquot.

10.3.5 Calculate the results according to the calculation section below.

NOTE 1: Low level alkalinity (<20mg/L) is performed by the autotitrator checking measurements every 0.3 pH units.

NOTE 2: If there is a pH greater than 4.5 with an alkalinity result of zero than the high level alkalinity method needs to be performed (see DV-WC-0085)

11.0 Calculations / Data Reduction

11.1 Standardization of Alkalinity Titrant

$$N_{ACID} = \frac{N_{BASE} \times V_{BASE}}{V_{ACID}}$$

Where:

N_{ACID} = Normality of titrant
N_{BASE} = Normality of Sodium Carbonate
V_{BASE} = Volume of Sodium Carbonate titrated, mL
V_{ACID} = Volume of titrant needed for titration, mL

11.2 pH values are recorded directly from the titrator printout.

11.3 Three values for alkalinity will be printed if the pH is greater than 8.3. Record the values at pH 8.3 and 4.5 on the bench sheet. The third value is the difference and is ignored. Note that some results will be printed in scientific notation; be careful to check this when recording results.

11.4 If the pH is less than 8.3, only one value will be printed (at pH 4.5). Record this value on the bench sheet. P alkalinity is ND on these samples.

11.5 Calculate the concentration as follows:

$$\text{Total Alkalinity, mg/L } CaCO_3 = \left(\frac{(2B - C) \times N \times 50{,}000}{\text{mL of sample}} \right)$$

Where:

B = Volume of titrant to first recorded pH, in mL.
C = Total volume of titrant added to reach a pH level 0.3 units lower.
N = Normality of acid.

11.6 Calculate the individual forms of alkalinity as follows:

Result of Titration	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2P	T - 2P
P = T/2	ND	2P	ND
P > T/2	2P - T	2(T - P)	ND
P = T	T	ND	ND

Where:

T = Total Alkalinity = Alkalinity at pH 4.5
P = Phenolphthalein Alkalinity = Alkalinity at pH 8.3

11.7 Record the values for Total, Bicarbonate, Carbonate, and Hydroxide Alkalinity.

11.8 Reporting

- Alkalinity results less than 5mg/L are reported as ND. All forms of alkalinity are reported in mg/L as Calcium Carbonate.
- Report case narratives for South Carolina must include date and time of collection.
- All data are subject to two levels of review, which is documented on the checklist shown in attachments 3 and 4.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.

- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows."

- 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

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, has the required experience, and has successfully analyzed initial demonstration samples (see SOP # DV-QA-0024 for details).

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Titrated sample waste – Aqueous Acidic - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 pH:

- Method 9040B, "pH Electrometric Measurement", Test Methods for Evaluating Solid Waste, EPA SW-846 Third Edition, 1/95.
- Method 4500-H+ B, pH Value, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

15.2 Alkalinity:

- Method 2320B, Alkalinity – Titration Method, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.
- U.S. EPA, Method for Chemical Analysis of Water and Waste, Method 310.1, Approved 1978.

16.0 Method Modifications:

Item	Method	Modification
1	310.1 & SM 2320B	The method requires an initial verification of the sulfuric acid titrant. The laboratory purchases the titrant from a commercial source and therefore does not perform additional verification.

17.0 Attachments

Attachment 1: Example Data Review Checklist for Titrations

18.0 Revision History

- Revision 6.2, dated 19 November 2010
  - Added reagent quality requirements to section 7.0
- Revision 6.1, dated 03 May 2010
  - Added section 6.3
  - Annual Review
- Revision 6, dated 27 March 2009
  - Deleted the Conductivity method from SOP
  - Updated other SOP references and formatting
  - Deleted Attachment 2 – Data Review Checklist for Direct Measurements.

- Added references to DV-WC-0085 (manual titration) for high alkalinity samples.

- Revision 5, dated 30 January 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting
  - Removed references to EPA method 305.1

- Revision 4.1, dated 20 November 2006
  - For this minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 4, dated 17 September 2002
  - Company name changed from Quanterra to STL Denver.
  - Definitions for conductivity and alkalinity were added to Section 3.
  - Details about the instrumentation were added to Section 6.
  - The pH meter is calibrated with 3 buffers, including a pH 7 buffer.
  - Details about the standards were added in section 7.
  - The old SOP made reference to LCS. Instead, this SOP refers to these solutions as calibration verification standards in Section 10.4
  - The high conductivity curve option was removed because current instrumentation does not allow adjustment of the cell constant.
  - The low level alkalinity option in the old SOP was redundant because all analyses use the 0.02N sulfuric acid, the low-level titrant.

Attachment 1.

Example Data Review Checklist for Titrations

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver Wet Chemistry Data Review Checklist
For Titration Methods

Test Name/Method #: _____ **Analysis Date:** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Was the normality of the titrant verified and found acceptable?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs/MDLs reflect required dilutions or limited sample volume?				
2. All reported results bracketed by in control LCS or QC Sample?				
3. Sample analyses done within holding time?				
4. Initial pH check documented for all samples (if required)?				
5. Preparation benchsheet completed and included in package (if applicable)?				
6. Special client requirements met?				
7. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
8. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
9. Are all data being reported highlighted on the benchsheet?				
10. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL for all reported samples > 10x method blank result?				
2. LCS run for batch and within QC limits?				
3. MS and/or MSD run at required frequency and within limits (if applicable)?				
4. Sample DUP run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Total Kjeldahl Nitrogen (TKN)

[EPA 351.2]

Approvals (Signature/Date):

David M. Elkin 2/11/10
Dave Elkin Date
Wet Chemistry Supervisor

2-11-10
Adam Alban Date
Health & Safety Manager / Coordinator

02/11/10
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 2/11/10
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure is used for the analysis of drinking and surface waters, domestic and industrial wastes, and soil samples for Total Kjeldahl Nitrogen (TKN), CAS# 5228003-90-0, by EPA Method 351.2.

1.2 During the digestion, amino acids, proteins, peptides and other nitrogenous compounds of biological origin are converted to ammonium sulfate. Nitrogenous compounds of some industrial waste such as amines, nitro compounds hydrazones, oximes, semicarbazones and some tertiary amines may not be converted.

1.3 The reporting limit is 0.50 mg/L for aqueous samples and 25 mg/kg for soil/waste samples. The range may be extended by sample dilution or by changing the detector sensitivity.

2.0 Summary of Method

Samples are digested by heating in the presence of sulfuric acid, potassium sulfate, and copper sulfate catalyst to a final temperature of 380°C. Free ammonia and organic nitrogen compounds are converted to ammonium sulfate. The ammonium is then reacted with salicylate and hypochlorite in a buffered alkaline solution in the presence sodium nitroprusside at a pH of 12.8 - 13.0 to form a blue-green compound whose intensity is measured at 660 nm.

3.0 Definitions

Total Kjeldahl Nitrogen (TKN) is the sum of free ammonia and organic nitrogen compounds.

4.0 Interferences

4.1 Some nitrogenous compounds such as amines, nitro compounds, hydrazones, oximes, and semicarbazones may not be broken down by the digestion procedure causing low TKN results.

4.2 During digestion, nitrate in excess of 10 mg/L can oxidize a portion of the ammonia released from the digested organic nitrogen, producing N_2O and resulting in a negative interference. When sufficient organic matter in a low state of oxidation is present, nitrate can be reduced to ammonia, resulting in a positive interference.

4.3 Inorganic salts and solids (from SM 4500-Norg A):
The acid and salt content of the Kjeldahl digestion reagent is intended to produce a digestion temperature of about 380°C. If the sample contains a very large quantity of salt or inorganic solids that dissolve during digestion, the temperature may rise above 400°C, at which point pyrolytic loss of nitrogen begins to occur. To prevent an excessive digestion temperature, add more H_2SO_4 to maintain the acid-salt balance. Not all salts cause precisely the same temperature rise, but adding 1 mL H_2SO_4/g salt in the sample gives reasonable results. Add the extra acid and the digestion reagent to both sample and reagent blank. Too much acid will lower the digestion temperature below 380°C and result in incomplete digestion and recovery. If necessary, add sodium hydroxide-sodium thiosulfate before loading onto the autoanalyzer to neutralize the excess acid.

4.4 The color reaction is very sensitive to the acid and salt content of the samples. All standards must be digested. Any dilutions performed after digestion must be made with the digested blank to maintain the proper matrix.

4.5 This procedure does not require distillation like some TKN determinations. See (67 FR 652198 – October 23, 2002) "Other TKN methods explicitly require alternate sample preparation procedures, such as the semi-automated block digestion (e.g., Method 351.2)."

4.6 Ammonia is included in the TKN result and is a common laboratory reagent and cleaning chemical. Contamination of samples with ammonia will give erroneously high results. Do not digest samples in the same hoods where ammonia is used as a reagent. Cleaning chemicals used in the laboratory should be ammonia-free. Glassware should be thoroughly rinsed with deionized water to remove any ammonia residues. After digestion, samples should be covered with foil in the tubes if they can not be promptly diluted and placed in capped vials.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.3 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Nitroferricyanide	Poison	5 mg/m^3 as HCN gas	Very toxic. May be fatal if inhaled or swallowed. May cause irritation in contact with the skin. **Gives off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.**
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Sever 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	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Block digestor (e.g. Tecator 2040 Digester with Controller)
- Alpkem (OI analytical) auto-analyzer system consisting of sampler, pump, manifold, detector, and computer with software WinFLOW for data system.

OR

- Astoria® Analyzer automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios. System consists of autosampler, multi-channel pump, TKN analysis cartridge, detector and computer with FASPac software.
- Pump tubing of appropriate size for use on Astoria® Analyzer or ALPKEM autoanalyzer.
- Nitrogen gas supply and appropriate lines and fittings

6.2 Supplies

- Thermometer (400°C range).
- Digestion tubes.
- Vortex mixer.
- Hengar boiling stones.
- Re-pipettor, 25 mL.
- Graduated cylinder, Class A, 25 mL
- Miscellaneous laboratory apparatus and glassware, including volumetric flasks, volumetric class A pipettes, beakers, etc.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

The automated salicylate method is utilized with this analysis; reagents vary depending on the instrument used. All reagents should be reagent grade unless manufactured specifically for this method. The reagents and their concentration listed below are for use with either the Alpkem autoanalyzer or Astoria autoanalyzer, as labeled.

7.1 Reagent water (ASTM type II or equivalent), distilled or deionized water, free of the analytes of interest.

7.2 **Digestion Reagents**
Dissolve 134 g of K_2SO_4 and 7.3 g of $CuSO_4$ in 800 mL of reagent water, **very slowly** and with constant stirring, add 134 mL of conc. H_2SO_4 acid. The solution will get **VERY HOT** on the addition of acid. Allow the solution to cool before diliuting to 1L in a volumetric flask. This reagent is also available commercially from RICCA Chemical.

7.3 **Manifold Startup Solution - Brij-35, 30% solution(ALPKEM or Astoria)**
Add approximately 1 mL Brij-35 (a solution purchased from commercial sources) to 500 mL of de-ionized water and mix gently.

7.4 **10 N Sodium Hydroxide (1 L)(ALPKEM or Astoria)**
Dissolve 250g of sodium hydroxide in approximately 800 mL of deionized water, allow to cool, then dilute with de-ionized water to 1L in a volumetric flask. Store tightly capped in a plastic container. Different volumes of 10 N sodium hydroxide can be prepared depending on requirement by increasing proportions. This reagent is available commercially from Fisher or VWR Scientific.

7.5 **Stock Buffer, Sodium Phosphate dibasic (1L)(ALPKEM or Astoria)**
Dissolve 134g of sodium phosphate-dibasic in approximately 800 mL of de-ionized water. Add 50 mL of 10N sodium hydroxide and dilute to 1L in a volumetric flask, mix well

7.6 **Stock Potassium Sodium Tartrate Solution (1L)(ALPKEM or Astoria)**
Dissolve 200g sodium potassium tartrate in approximately 800 mL of de-ionized water and dilute to volume in a 1L volumetric flask. Dilute to mark and mix well.

7.7 **Working Buffer (1L)(ALPKEM)**
Add 200 mL of stock phosphate buffer (7.5) to 200 mL of de-ionized water in a 1 L volumetric flask and mix. While stirring, add 250 mL of stock potassium sodium tartrate (7.6). Continue stirring and slowly add 60 mL of 10 N sodium hydroxide (7.4). Dilute the solution to the mark and mix well. Degas the solution, then add 0.5 mL of Brij-35, 30% and mix gently to prevent foaming. Degas the solution prior to use.

7.8 **Working Buffer (1L)(Astoria)**
Add 200 mL of stock phosphate buffer (7.5) to 200 mL of de-ionized water in a 1 L volumetric flask and mix. While stirring, add 250 mL of stock potassium sodium tartrate (7.6). Continue stirring and slowly add 88 mL of 10 N sodium hydroxide

(7.4). Dilute the solution to the mark and mix well. Degas the solution, then add 10 drops of of Brij-35, 30% and mix gently to to prevent foaming. Filter if baseline is noisy.

7.9 Sodium Salicylate/Sodium Nitroprusside Solution(500 mL)(ALPKEM or Astoria)

Dissolve 75g of sodium salicylate and 0.3g of sodium nitroferricynide in approximately 300 mL of de-ionized water and dilute to volume with deionized water in a 500 mL volumetric flask. Mix well to completely dissolve all sodium nitroprusside. This solution must be stored in a dark (light-resistant) container.

7.10 Sodium Hypochlorite Solution(ALPKEM or Astoria)

Add 12 mL of sodium hypochlorite solution to approximately 180 mL of de-ionized water in 200 mL volumetric flask. Dilute to mark and mix well. Prepare fresh daily from commercial liquid bleach. Do not use "ultra" or scented bleach. Store in an amber bottle.

7.11 Calibration Standards

7.11.1 Ammonia Cal Stock Standard, 1000 mg/L as N

Dry ammonia chloride at 105 C. Dissolve 3.819 g in 5600 mL of deionized wate, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also.

7.11.2 Ammonia Cal Intermediate Cal Standard, 100 mg/L as N

Add 50 mL of Ammonia Cal standard to a 500 mL which also contains 1.0 mL of 1:1 sulfuric acid. Dilute to the mark with deionized water.

7.11.3 Ammonia Cal Intermediate Cal Standard, 25 mg/L as N

Add 25 mL of Ammonia cal intermediate standard to a 100 mL which also contains 1.0 mL of 1:1 sulfuric acid. Dilute to the mark with deionized water.

7.11.4 Second-Source Calibration Verification Standard (ICV)

Purchase a commercially prepared concentrate with a certified value equal to or greater than 2 mg/L. This should be prepared according to manufacturer's instructions and has a true value that varies with individual lot of standard. Normally, ERA Complex Nutrients is the ICV, but equivalent standards may be used.

7.11.5 Calibration Standards

All standards must be digested. New standards must be prepared and digested at least monthly. Store digested standards in tightly sealed containers to prevent absorption of ammonia. Alternatively, these can be prepared by spiking directly into digestion tubes.

Prepare standards as indicated below using the 25 mg/L intermediate stock:

Volume (mL) Standard (7.9.1)	Final Volume (mL)	Concentration as mg/L N
0.25	25	0.25
0.5	25	0.5
1.0	25	1.0
2.0	25	2.0
*5.0	25	5.0
10.0	25	10.0

*This level is prepared as the Continuing Calibration Verification (CCV).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE or Glass	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soils	Glass	4 oz	Cool 4 ± 2°C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and

trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Method Blank

A method blank (de-ionized water) is required with every batch of 20 or less samples.

Acceptance Criteria: Method blanks must be less than two times the reporting limit.

Corrective Action: If this level is exceeded, the batch must be reprepared and reanalyzed.

9.1.2 Laboratory Control Samples (LCS)

An LCS is required with every batch of 20 or less samples. The LCS standard is 6.0 mg/L made from the 6.0 mL of intermediate stock standard diluted to 25 mL with de-ionized water.

Acceptance Criteria: LCS control limits are based on historical data (see Policy DV-QA-003P for details), and the control limits are available in LIMS.

Corrective Action: If the acceptance criteria are not met, the batch must be reprepared and reanalyzed.

9.1.3 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

An MS and MSD are required with every 10 samples or less. Add 3 mL of 25 mg/L standard to 25 mL of sample and digest. The spiking concentration is 3 mg/L.

Acceptance Criteria: MS/MSD recovery and relative percent difference control limits are based on historical data, and the control limits are available in LIMS.

Corrective Action: If MSD/MSD recoveries exceed allowable levels and the LCS is in control, the data will be flagged as outside of control limits. Document the results in an exception report. If the RPD is greater than RPD limit the samples should be reanalyzed.

9.1.4 Troubleshooting Guide

- Check for contamination in the reagents and standards. Be sure all reagents were made correctly and have not exceeded their expiration dates.
- All samples and standards must contain the same salt and acid concentrations.
- Check system for obvious problems such as plugs and leaks.
- Low LCS recovery usually indicates incomplete digestion.
- If you are unable to locate and solve the problem, consult your supervisor.

9.2 Instrument QC

9.2.1 Initial calibration verification standard (ICV): Immediately following the initial calibration, a mid-range second-source initial calibration verification standard (ICV) is analyzed. The recovery for this standard must be within 10% of the true value. If this is not achieved, the instrument must be recalibrated.

9.2.2 Continuing calibration verification standard (CCV): A CCV is analyzed at a frequency of at least 10% between samples, and analyzed at the end of the analytical sequence. The acceptance criteria for the CCV is ± 10% of the true value. If this is not achieved, the instrument must be recalibrated and the samples run since the last successful CCV must be reanalyzed.

9.2.3 Continuing calibration blank (CCB): A CCB is analyzed after every CCV. The CCB consist of deionized water. The CCB results must be less than the reporting limit. If the blank is greater than the reporting limit, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.

9.2.4 Linear Calibration Range (LCR): The LCR is performed initially by each analyst and then verified every analysis. A blank, LCS, CCV, and ICV are performed each analysis, this fulfills the method requirement of a blank and three standards for verification of linearity.

Acceptance Limits: The acceptance limits for the LCR standards are ± 10% of the true value.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, and chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Interference Checks

The measuring and recording the pH of water samples with pH paper is done at the time of receipt of the samples in sample receiving. The pH should be less than 2. If it is not, prepare a nonconformance describing the problem and check with the lab PM before proceeding.

10.2 Sample Preparation(ALPKEM or Astoria)

10.2.1 Prepare a block digestor lay-out sheet to determine what will be loaded into each digestion tube position.

10.2.2 Place 25 mL of sample, blank, LCS and calibration standards into the appropriate digestion tubes. For calibration standards and LCS, pipette the appropriate amount of stock or intermediate standard to each tube and rinse it down the side well with de-ionized water to an approximate total volume of 25 mL. Use volumetric class A pipettes or micro-liter pipettes.

10.2.3 For solid samples, weigh 0.5 g sample into the tube and rinse down any sample that clings to the side walls of the tube using deionized water.

10.2.4 Samples which contain high levels of salt or organic nitrogen may need to be diluted prior to digestion. Record any dilutions performed on the bench sheet.

10.2.5 Add 5 mL of digestion reagent.

10.2.6 Add 4-6 Hengar Granules and place the tubes on the block digestor. Use a digestor lay-out sheet to record the sample IDs located in each position.

10.2.7 Heat the samples for 2 hours at 140 °C followed by 2 1/2 hours at 380 °C. Depending on the model used, it may be possible to temperature program the block digestor. See the block digestor manual for specific programming instructions.

10.2.8 After digestion, allow the tubes to cool to room temperature.

10.2.9 Carefully and accurately add 25mL of de-ionized water using a class A graduated cylinder to measure the water. Point the tube toward the back of the hood when doing this in case the mixture splatters out of the tube. Samples may be allowed to sit for a short time with the deionized water in order to aid in dissolution of the salts before proceeding to 10.2.10.

10.2.10 Mix the samples on the vortex mixer and ensure that all salts in the bottom of the tubes have completely dissolved. Transfer to a labeled vial and seal. Analyze the samples as soon as possible after digestion.

10.3 System Startup: ALPKEM

NOTE: Consult the Alpkem operating instructions located near the instrument for guidance on start up. This method is somewhat generalized - the specific detail for analysis on the Alpkem Auto-Analyzer systems can be found in operation manual on line.

10.3.1 Be sure that the instrument is set up with the correct manifold, flow cell, detector wavelength (660 nm), temperature, etc. A generalized flow diagram is attached (Attachment 1); detailed information can be found in the instructions for the specific instrument being used. The temperature on the TKN heating coil should be set to 37°C.

10.3.2 Check reagent levels and replenish as needed.

10.3.3 Allow the sytem to flow with start-up solution (7.3) for approximately 10-15 minutes.

10.3.4 Once the flow is stable, place the reagent lines into the reagent containers, except for the salicylate line. **CAUTION:** If the salicylate reagent is pumped too soon, a precipitate may form inside the lines.

10.3.5 The salicylate line may be placed in its proper container after the other reagents have been pumped through the entire system.

NOTE: If a precipitate forms after the addition of salicylate, immediately stop the proportioning pump and flush the coils with water using a syringe. Precipitation of salicylic acid is caused by a low pH. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution.

10.4 System Startup: Astoria

NOTE: Consult the Astoria operating manual located near the instrument for guidance on start up. This method is somewhat generalized.

10.4.1 Be sure that the instrument is set up with the correct manifold as shown in the flow diagram included in Attachment 2. The temperature on the TKN heating coil should be set to 50°C.

10.4.2 Turn on the power to all units including heating coil and place all reagent lines in startup solution.

10.4.3 Latch the pump platens on both the main and auxiliary (traveling wash reservoir) pumps and begin flow. Verify that the bubble size and spacing is consistent.

10.4.4 Allow the sytem to flow with start-up solution (7.3) for approximately 10-15 minutes.

10.4.5 Once the flow is stable, place the reagent lines into the reagent containers, except for the salicylate line. **CAUTION:** If the salicylate reagent is pumped too soon, a precipitate may form inside the lines.

10.4.6 The salicylate line may be placed in its proper container after the other reagents have been pumped through the entire system.

NOTE: If a precipitate forms after the addition of salicylate, immediately stop the proportioning pump and flush the coils with water using a syringe. Precipitation of salicylic acid is caused by a low pH. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution and make sure all lines are pumping properly. Check reagents for any contamination caused by backflow when the manifold was clogged.

10.5 Calibration(ALPKEM or Astoria)

Calibration standards are prepared as described in Section 7.11, and digested as described in Section 10.2. The instrument start up is also described in Section 10.3. Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

The instrument is initially calibrated (ICAL) using the six concentrations shown in 7.9. The correlation coefficient r for the linear regression equation should be 0.995 or more. If the r is less than 0.995, investigate and correct problem before recalibrating.

10.6 Sample Analysis(ALPKEM or Astoria)

10.6.1 Prepare the sample table with the appropriate calibration and with the intended sequence of samples.

10.6.2 Load the calibration standards and samples into the autosampler.

10.6.3 Initiate the autosampler and start with the analysis of the sync peak. Verify that an acceptable calibration curve has been obtained before proceeding.

10.6.4 The ICV, ICB, LCS and MB should be analyzed next.

10.6.5 Analyze samples. Any samples which exceed the response of the high standard must be diluted with digested blank solution and reanalyzed. Samples must not be diluted with deionized water but with digested blank solution, or an incorrect value will be reported. Do not over-dilute the samples.

10.6.6 Analyze a continuing calibration verification and continuing calibration blank after every 10 or less samples, and again at the end of the run.

10.6.7 Carefully monitor the reagent to ensure that you do not run out of buffer. Running out of buffer will cause salicylate to precipitate out in the lines.

10.7 Instrument Shut-Down(ALPKEM)

10.7.1 Remove the salicylate line first and place in start-up solution. Allow reagents to flow for several minutes until all salicylate has been flushed from the system.

10.7.2 Place all other reagent lines in start-up solution and flush the system for at least 10 minutes.

10.7.3 Turn off the instrument modules and release the pump platens.

10.8 Instrument Shut-Down(Astoria)

10.8.1 Remove the salicylate line first and place in start-up solution. Allow reagents to flow for several minutes until all salicylate has been flushed from the system. Turn off the heating coil.

10.8.2 Place all other reagent lines in start-up solution and flush the system for at least 10 to 15 minutes.

10.8.3 Turn off the instrument modules and release the pump platens from both the main and auxiliary pumps.

11.0 Calculations / Data Reduction

NOTE: The Alpkem and Astoria systems automatically perform all data acquisition.

11.1 Enter the standard concentrations and peak heights into a linear least squares program. The Alpkem software performs these calculations automatically.

11.2 All calibration equations can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005. Use the least squares equation to calculate the results for the samples from the peak heights. Multiply the result obtained by any dilutions made during prep or analysis. The Alpkem software does this automatically, so long as the dilution factor is entered into the dilution factor field in the sample table.

11.3 Total Organic Nitrogen can be determined by subtracting ammonia nitrogen from the TKN result. TKN and ammonia are often analyzed on the same sample. The TKN result should always be greater than or equal to the ammonia result, taking into account the possible analytical errors.

11.4 Reporting

- Reporting units are mg/L as N for aqueous samples and mg/kg as N for soil/waste samples.
- Results less than the reporting limit is reported as ND. The detection limits must be raised if dilutions were made during sample prep or due to interferences.
- All results are subject to two levels of data review, which are documented on the checklist shown in Attachment 3.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Four aliquots of the QC check sample (independent source from the calibration) 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 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. Further details concerning the training program are described in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acid Waste (Wet Chemistry) - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method Source: EPA Method 351.2, Revision 2.0, August, 1993

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 4500-Norg A

15.3 Related Documents

- Tecator Manual, 2000 Digestion System
- Alpkem (OI Analytical) Automated Ion Analyzer Manual

16.0 Method Modifications:

Item	Method	Modification
1	EPA 351.2	Method 351.2 states the calibration standards are made with ammonium chloride. The laboratory uses L-glutamic acid to calibrate for verification of the digestion process. A series of three ammonium chloride standards were prepared at levels of 5 ppm and 0.2 ppm, and then analyzed against the L-glutamic acid curve. The standards were found to be comparable to the organic nitrogen standards at the 5 ppm level with ± 10% and ± 50% at the MDL concentration. The results are kept on file for proof of verification.
2	EPA 351.2	The initial temperature for digestion was reduced to 140°C to prevent overheating and splattering of the samples, which occurred at the higher temperature (160°C) specified in the source method. The time was increased to be sure that the water is completely driven off before ramping to the high temperature.
3	EPA 351.2	The dynamic range has been changed to 0.25 - 7 mg/L from 0.1 - 20 mg/L to improve linearity.

17.0 Attachments

Attachment 1: Flow Chart
Attachment 2: Manifold Flow Diagram
Attachment 3: Example Data Review Checklist

18.0 Revision History

- Revision 6, dated 12 February 2010
  - Added Section 6.3
  - Updated Sections 7.11.1 to 7.11.3
  - Updated Section 7.11.5 (standards table)
  - Updated Section 9.1.2

- Revision 5, dated 24 July 2009
  - Fixed multiple section references throughout SOP
  - Deleted Attachment 1 Digestor instructions
  - Added reference to the digestor manual to section 10.2.7
  - Added Attachment 2 Manifold Flow diagram
  - Updated Attachment 3 to current checklist

- Revision 4, dated 04 May 2009
  - Added the Linear Calibration Range information in Section 9.2.4
  - Added method modification #1
  - Clarified that IDOCs need to be performed with a second source standard.
  - Corrected minor grammatical and formatting errors.

- Revision 3, dated 30 September 2008
  - Added Astoria technical information throughout SOP.

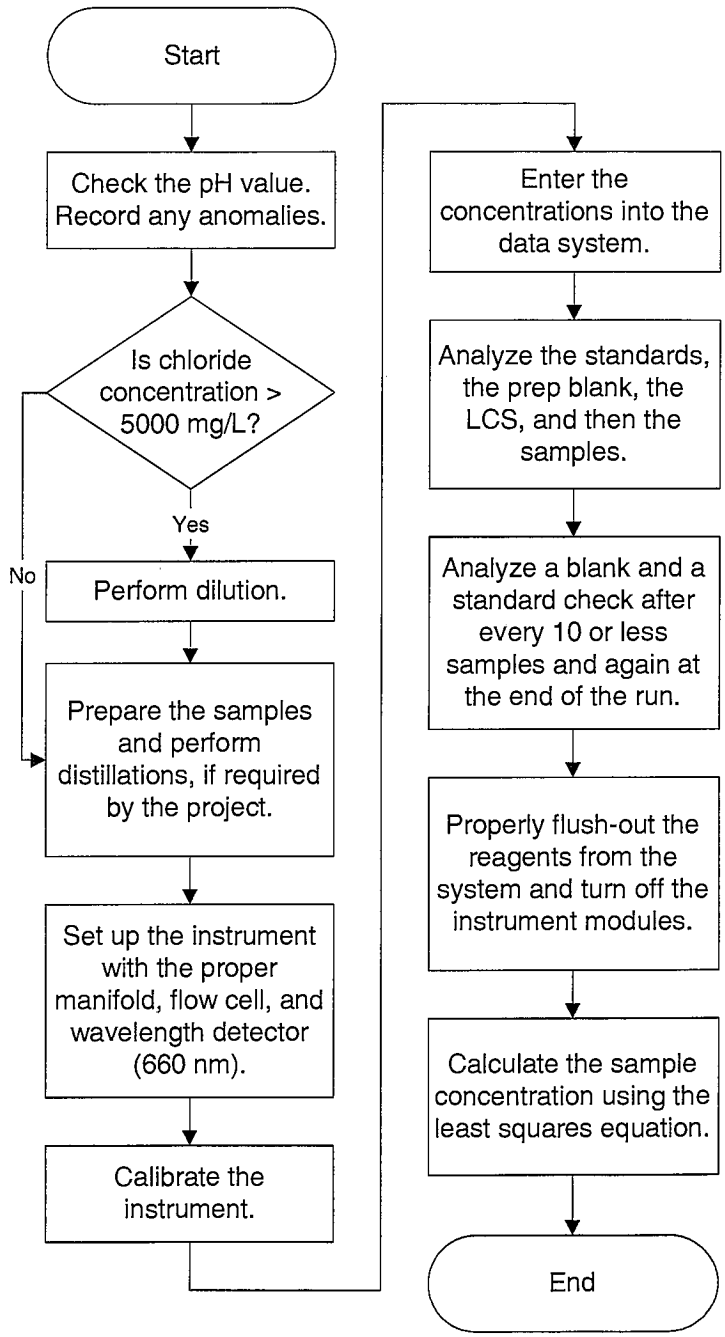
- Revision 2, dated 29 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting
  - Changed the nomenclature for SOPs throughout the SOP to match the new format.

- Revision 1.1, dated 20 November 2006
  - For this minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 1.0, dated 15 January 2003
  - Safety and Waste Management sections were updated to reflect current format and practice.
  - Initial calibration levels were updated.
  - A reference to a distillation for New Jersey was removed, and a citation noting that distillation is not part of this procedure was added as section 4.5.
  - An example Data Review Checklist was added as Attachment 2.
  - Section 13, Method Performance, was updated to reflect current content and format.

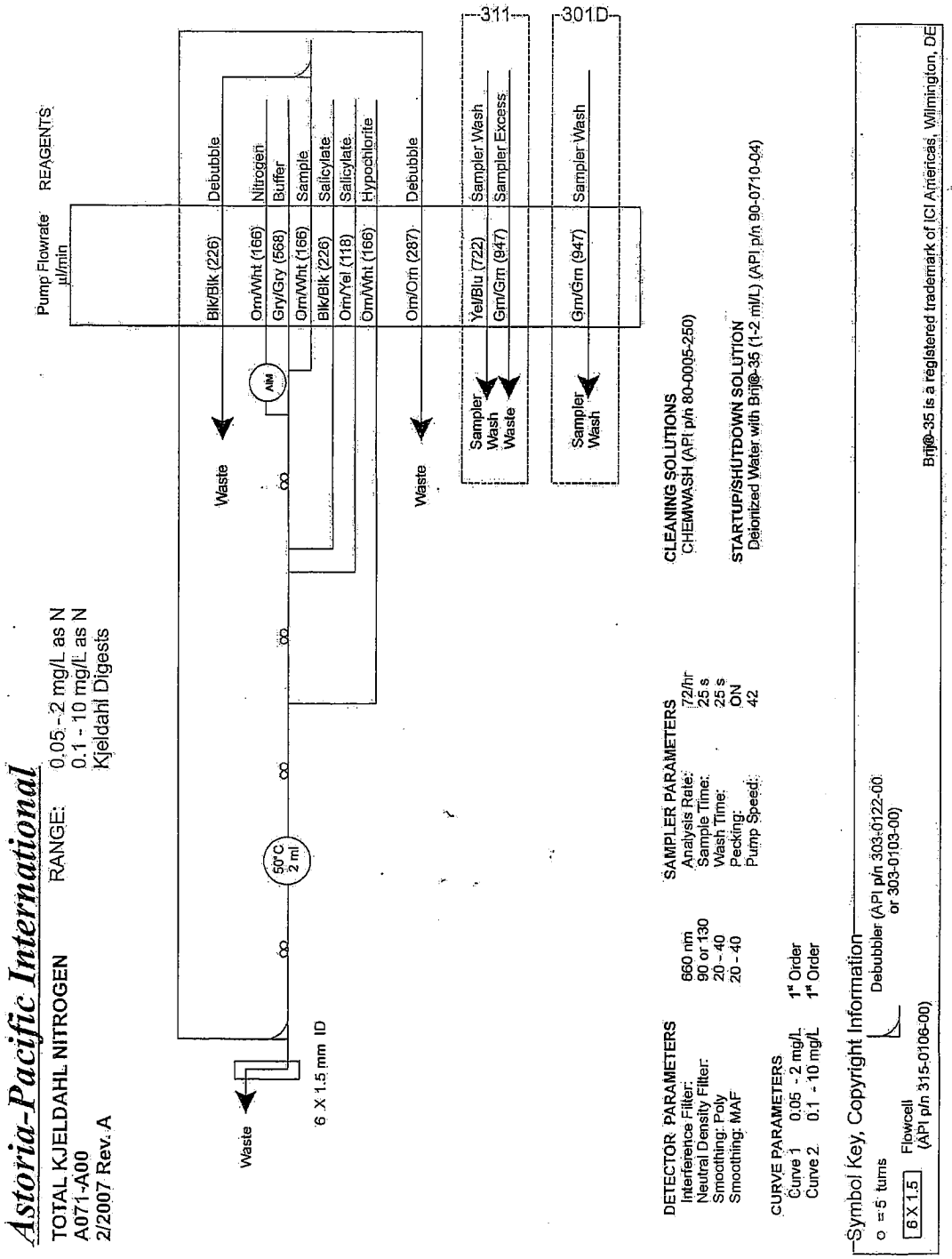
Attachment 1.

Flow Chart



Attachment 2.

Manifold Flow Diagram



Attachment 3.

Example Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Inst

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. Are reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lots/Dates report checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD/True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

TestAmerica
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-Title: Ammonia Nitrogen by Autoanalyzer [EPA 350.1]

Approvals (Signature/Date):

Dave Elkin — Date 12/21/10
Wet Chemistry Supervisor

Adam Alban — Date 21 Dec 10
Health & Safety Manager / Coordinator

John P. Morris — Date 12/21/10
Quality Assurance Manager

Robert C. Hanisch — Date 12/21/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This procedure describes the automated phenolate analysis of water samples for ammonia by EPA Method 350.1.

1.2 The reporting limit for ammonia is 0.1 mg/L.

1.3 This method covers the determination of ammonia in drinking, ground, surface, and saline water, domestic and industrial wastes.

2.0 Summary of Method

2.1 The automated phenolate method involves the reaction of alkaline phenol and hypochlorite with ammonia to form indophenol blue. The color is enhanced by the addition of sodium nitroprusside.

2.2 The intensity of the color at 660nm is proportional to the ammonia concentration.

3.0 Definitions

There are no terms requiring definition unique to this procedure. Refer to the Glossary of the QAM for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Residual chlorine will react with ammonia to form chloramines. Residual chlorine must be removed at the time of sample collection by pretreatment of the sample with sodium thiosulfate or other reagent before distillation.

4.2 Wastewater discharge monitoring and other Clean Water Act compliance testing requires either distillation or demonstration of equivalency between distilled and undistilled samples. If significant interferences are encountered, a preliminary distillation may be necessary.

4.3 Calcium and magnesium may precipitate as hydroxides or carbonates in alkaline solutions and clog the ammonia channel or cause turbidity problems. EDTA is added to minimize this effect.

4.4 Color and turbidity in the samples will interfere. If color or turbidity are observed the sample is filtered through GFC filter media. If color remains after filtration, the sample is diluted until the color absorption at 660nm is minimal.

4.5 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.

4.6 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that biases analytical response.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Sodium nitroferricyanide will generate hyrdogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.

5.1.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Chloride	Corrosive	10 mg/m^3 (TWA)	Causes irritation to respiratory tract; symptoms may include coughing, shortness of breath. Ingestion may cause nausea, vomiting, and diarrhea. Causes irritation to skin and eyes.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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.
Sodium Nitroferricyanide	Poison	5 mg/m3 as HCN gas	May cause irritation in contact with the skin. Gives off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.
Phenol	Corrosive	5 ppm (TWA)	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
EDTA	Flammable	None	Mild irritant to respiratory tract. Symptoms may include coughing and sneezing. Low toxicity by ingestion. Prolonged contact with skin may cause redness or inflammation.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Auto-analyzer equipped with the proportioning pump, autosampler, and colorimeter with 660 nm filters and 5-mm flow cell.
- Analytical balance, capable of accurately weighing to the nearest 0.0001 g. The accuracy of the balance is verified each day it is used in accordance with SOP DV-QA-0014.
- An all glass distilling apparatus (Kontes or WESTCo).

6.2 Supplies

- Volumetric Flasks (Class A): varying volumes
- Eppendorf Pipettes, varying volumes

6.3 Computer Software and Hardware

Please refer to the master list of documents and software/hardware located on G\QA\Read\Master List of Documents\Master List of Documents and Software/hardware.xls

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Reagent Water
Reagent grade deionized water, Milli-Q or equivalent. The water must have less than 0.1 mg/L ammonia.

7.2 Sulfuric Acid (H_2SO_4), 0.04 N
Dilute 1.0 mL of concentrated H_2SO_4 to 1 liter. This is the distillation collection solution.

7.3 Sulfuric Acid (H_2SO_4), 5 N:
Carefully add 139 mL of concentrated sulfuric acid to approximately 500 mL of reagent water. Cool to room temperature and dilute to 1 liter with deionized water. This is the air scrubber solution used in the autoanalyzer.

7.4 Sulfuric Acid, 0.2%
Slowly and with constant mixing add 4 mL concentrated sulfuric acid to 2 liters of de-ionized water.

7.5 Sodium Phenolate
Add 80 mL of 10 N sodium hydroxide to 700 mL of reagent water. Slowly add 83g of phenol (94 mL of liquid phenol) in small amounts. Bring to a final volume of 1 liter with deionized water. Store in the dark and prepare fresh every week.

7.6 Sodium Hypochlorite Solution (NaOCl)
Dilute 250 mL of a bleach solution containing 5.25% NaOCl (such as Clorox) to 500 mL with deionized water. Analysts must remain alert to detecting any variation in the formulation as the percentage of NaOCl changes in Clorox. Due to the instability of this product, solution should be prepared fresh daily.

7.7 Sodium Nitroferricyanide Solution
Dissolve 0.5 g of sodium nitroferricyanide in 1 liter of deionized water. Store in an amber container and prepare fresh monthly.

7.8 Dechlorinating reagents;

7.8.1 Sodium Thiosulfate ($Na_2S_2O_3$)
Dissolve 3.5g of $Na_2S_2O_3 \cdot 5H2O$ in reagent water and dilute to 1 liter.

7.8.2 Sodium Sulfite (Na_2SO_3)
Dissolve 0.9g of Na_2SO_3 in reagent water and dilute to 1 liter.

7.9 Brij-35 Solution
This solution is obtained from commercial sources.

7.10 Borate Buffer
Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025 M sodium tetraborate solution (5 g anhydrous $Na_2B_4O_7$ or 9.5 g of $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute with reagent water. This solution is also available from commercial sources.

7.11 Sodium Hydroxide Solution, 1.0 N
Reagent grade solution is available from commercial sources. Alternatively, it is prepared by dissolving 40g of NaOH in reagent water and diluting to 1 liter.

7.12 Disodium EDTA
Add 50 g of disodium EDTA and 6 pellets of sodium hydroxide to 800mL of deionized water. Cool to room temperature and bring to a final volume of 1 liter. Add 20 drops or 1 mL of Brij-35 and mix.

7.13 Ammonia CAL Stock Standard, 1000mg/L
Dry ammonium chloride at 105 °C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also.

7.14 Ammonia ICV Stock Standard, 1000 mg/L
Dry ammonium chloride at 105 °C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also. This solution should be prepared from a separate source from that used for the CAL standard.

7.15 Intermediate CAL Standard, 100 mg/L
Pipette 10 mL of stock standard into a 100 mL volumetric flask, dilute to volume with distilled water and add 2 drops concentrated sulfuric acid.. This standard can also be purchased from a commercial vendor.

7.16 Intermediate ICV Standard, 100 mg/L
Pipette 10 mL of stock standard into a 100 mL volumetric flask, dilute to volume with distilled water, and add 2 drops of concentrated sulfuric acid. This standard can also be purchased from a vendor.

7.17 Working Calibration Standards
Prepare dilutions as indicated below. These dilutions can be made by the auto-analyzer system. All dilutions are made with distilled water and are remade daily. The following table shows suggested concentrations:

Volume of Intermediate Standard (mL)	Final Volume (mL)	Concentration (mg/L as N)
0.05	100	0.05
0.10	100	0.10
0.50	100	0.50
1.0	100	1.0
5.0	100	5.0
10.0	100	10.0

NOTE: If nitrate/nitrite is being run at the same time as ammonia, a mixed intermediate and mixed calibration standards may be prepared that contain both ammonia and nitrate at the above concentrations.

7.18 Initial Calibration Verification Standard (ICV)
This is a second-source standard, different vendor than the calibration standards. It is prepared at a concentration of 2.0 mg/L by pipetting 2.0 mL of the 100 mg/L intermediate ICV standard and diluting to 100 mL with distilled water. This solution must be remade daily.

7.19 Laboratory Control Sample (LCS)
The LCS is identical to the 5.0 mg/L standard prepared as a working calibration standard. The LCS for soil samples is prepared by adding 1 mL of the Cal Stock Standard 1000 mg/L (7.13) to 10 g of solid matrix (Ottawa sand, boiling chips, glass beads, etc.).

7.20 Matrix spike/matrix spike duplicate (MS/SD)
The Matrix Spike and Matrix Spike Duplicate (MS/MSD) samples are prepared by adding 0.20 mL of the Intermediate CAL Standard, 100 mg/L(7.15) to 4.8 mL of sample. The true value of the spike is 4.0 mg/L. The MS/MSD for soil samples is prepared by adding 1 mL of the Cal Stock Standard, 1000 mg/L (7.13) to 10 g of sample.

7.21 Kleenflow Solution - Acidic
Add 50 mL of Isopropyl Alcohol to a 1 liter flask. Carefully add approximately 950 mL of 1N Hydrochloric Acid (HCL).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soil	Glass	4 oz	Cool 4 ± 2°C	28 Days	N/A

NOTE: Samples collected for ammonia nitrogen must be dechlorinated in the field. 1 mL of sodium thiosulfate or sodium sulfite solution removes 1 mg/L of residual chlorine per 500 mL of sample. If necessary, neutralize to approximately pH 7 with dilute acid or base, using a pH meter.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

9.1.1 Method Blank

A method blank of deionized water is required with every batch of 20 or fewer samples. The MB consists of reagent water that is carried through the entire analytical procedure, including preparation and analysis. When analyzing soils, the MB is prepared in the same manner as the samples by a solid matrix (Ottawa sand, boiling chips, glass beads, etc.). The MB 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.

Acceptance Criteria: The blank result must be < ½ RL, although samples more than ten times the blank concentration may be reported with a footnote or narrative comment.

Corrective Action: If the blank criterion is not met, the source of the contamination should be investigated and corrected. All associated samples should be reanalyzed or the data evaluated and reported with the appropriate qualifiers.

9.1.2 Laboratory Control Sample (LCS)

One LCS sample is required for each batch of 20 or fewer samples. The LCS for aqueous sample batches consists of reagent water to which a known amount of target analyte has been added. The LCS for soil batches consists of 10 grams of a solid matrix (Ottawa sand, boiling chips, glass beads, etc.)

to which a known amount of the target analyte is added. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Preparation of the LCS is described in Section 7.19.

Acceptance Criteria: The recovery for the LCS must be within the laboratory's statistical (three standard deviations) control limits, not wider than 90-110%.

Corrective Action: Samples analyzed along with an LCS that is determined to be "out of control" are considered suspect and the samples must be reprocessed and reanalyzed, or the data reported with appropriate data qualification. If the LCS result does not fall within statistical control limits, check calculations, check instrument performance, reanalyze the LCS, and if still outside of control limits, re-prepare and reanalyze all samples in the QC batch. It is acceptable to report the data if the LCS recovery is out high and any analyte of concern was not detected in any of the samples.

9.1.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Samples

An MS/MSD pair is required for every 10 or fewer samples. Preparation of matrix spikes and matrix spike duplicates is described in Section 7.20.

Acceptance Criteria: The recovery for each matrix spike must be within the laboratory's statistical (three standard deviations) control limits, not wider than 90-110%. The relative percent difference (RPD) for the MS/MSD pair must be within 10%.

Corrective Action: If these limits are not attained, associated sample results must be qualified on the final report. Check special program or project requirements on this point because some require additional corrective actions.

NOTE: Some programs (North and South Carolina) require spiking at 10% rate and a spike duplicate is not sufficient for these programs. Refer to client requirement for these special instructions.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV)

The result for a second-source calibration verification standard must be within ± 10% of the expected value. If this is not attained, reprepare the initial calibration standards and/or optimize the instrument and recalibrate.

9.2.2 Continuing Calibration Verification (CCV)

A mid-point calibration standard is analyzed after every ten samples and at the end of the run. The results for the CCVs must be within ± 10% of the expected value. If routine corrective action fails to produce a second consecutive (immediate) CCV within acceptance limits, than two consecutive successful CCVs need to be performed or the instrument should be recalibrated and all samples tested since the last successful CCV reanalyzed.

NOTE: If a quadratic curve is used 2 CCV concentrations must be performed every 10 samples. The concentrations used will be 1.0 mg/L and 5.0 mg/L.

9.2.3 Continuing Calibration Blank (CCB)
A deionized water blank is analyzed after every ten samples and at the end of the run. The results for the CCBs must be less than 0.1 µg/L. If the CCB results are over this limit, maintenance must be performed to decontaminate the system and all samples run since the last successful CCB reanalyzed.

9.2.4 Linear Calibration Range (LCR)
The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, the linearity must be re-established.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 The pH of all samples are checked in log-in at the time of receipt. If the pH is above 2, results may not be acceptable – A Condition Upon Receipt (CUR) form is completed to notify the laboratory Project Manager. Because results for improperly preserved samples may be biased low, analysis should not proceed without authorization and the final report must qualify the result.

10.1.2 The laboratory **does not** distill all water samples for Method 350.1, but will perform as requested by the client. If distillation is requested the sample will be logged with the distillation prep code.

10.1.3 The laboratory distills **all** soil samples for method 350.1.

10.1.4 Sample Distillation

10.1.4.1 Test sample for residual chlorine with KI paper. If residual chlorine is detected, it means that the sample was not collected properly and the laboratory Project Manager must be notified. The client should then be consulted to determine how to proceed. If the client gives the order to proceed, an NCM must be prepared and the final report case narrative must describe the problem.

10.1.4.2 Add 8 mL of borate buffer to the sample adjusted to pH 9.5 and transfer to a 500 mL distillation flask

10.1.4.3 Add 1 N NaOH, until the pH is 9.5. Check the pH during the addition with a pH meter or by use of a short-range pH paper.

10.1.4.4 Place 200 mL of sample in a distillation flask and set up distillation apparatus.

10.1.4.5 For soil samples, place 10 g of soil in a distillation flask, add 200 mLs DI water and set up distillation apparatus. Then add the borate buffer (Section 7.10) until the pH is 9.5

10.1.4.6 Distill at a rate of 6-10 mL/min. Distill ammonia into 50 mL of 0.04 N H2SO4. Collect at least 100 mL of distillate. Dilute distillate to 200 mL with reagent water.

10.2 Calibration

10.2.1 Instrument Set-up

Follow the specific instrument set up directions from the equipment vendor.

10.2.2 Initial Calibration

The instrument is calibrated at six concentration levels from 0.05 to 10.0 mg/L (see Section 7.17). A calibration curve is constructed using a linear least squares regression. A minimum correlation coefficient of $r \geq 0.995$ is required. If this is not met, reprepare standards and/or optimize the instrument and recalibrate.

10.2.3 If a linear squares regression is not the best curve fit, alternatively a quadratic curve fit may be used (2^{nd} order curve). The instrument software uses a quadratic regression to relate the concentration of ammonia in each standard and the associated absorbance reading, as follows:

$$x = cyy + by + a \qquad \textbf{Equation 1}$$

Where:

x = *analyte concentration*
y = *analyte height*
c = *definition of the curve*
b = *Slope.*
a = *Y-intercept.*

10.2.4 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Samples are loaded on the autosampler, processed. The instrument data system prints a report of results calculated in mg/L.

10.3.2 Samples with results exceeding the highest calibration standard are diluted and rerun. Appropriate dilutions produce results in the upper one-half of the calibration range.

10.3.3 Following is a typical analytical sequence:

ICAL and/or ICV and ICB

LCS and LCSD
Method Blank
7 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB
10 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB
10 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB

10.3.4 Disconnect the reagent lines and put all of them into DI water, EXCEPT the buffer line. The buffer line should be placed into bridge solution.

10.3.5 Rinse all tubes for at least 5 minutes, and then rinse with Kleenflow Base for 5 minutes. Rinse all tubes with the appropriate rinses for another 5 minutes.

10.3.6 Turn off the instrument and pump, rinse platens, and empty waste when finished.

11.0 Calculations / Data Reduction

11.1 Accuracy

ICV / CCV, LCS % Recovery = observed concentration x 100

known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100

spiked concentration

11.2 Precision (RPD)

Matrix Duplicate (MD) = |orig. sample value - dup. sample value| x 100

[(orig. sample value + dup. sample value)/2]

11.3 Concentration

$$\text{Ammonia (as mg/L N)} = \frac{(C \times DF)}{V}$$

Where:

C = Concentration measured at the instrument
DF = Dilution factor
V = Volume of sample used for analysis

- Normally this calculation is performed automatically by the instrument data system.

- If an extract of a non-aqueous sample was analyzed, multiply the result obtained by the prep dilution factor to obtain the final result.

11.4 See Attachment 1, "Wet Chemistry Review Checklist for Tests with Calibration Curves," for first and second-level data review documentation requirements.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Initially the analyst must perform an MDL study (see section 12.1).
- Four aliquots of the QC check sample (independent source from the calibration) 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Distillation waste – Aqueous Alkaline - Waste Stream E
- Distillate and Instrument Waste – Aqueous Acidic - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 EPA 350.1, Determination of Ammonia Nitrogen by Semi-Automated Colorimetry, Revision 2.0, August 1993

15.2 Alpkem manual for Enviroflow 3500 (P/N 000156 & P/NO 00157)

16.0 Method Modifications:

Item	Method	Modification
1	EPA 350.1	Method 350.1 requires all samples to be distilled. The laboratory only distills samples as requested by the client.
2	EPA 350.1	Method 350.1 states to place the calibration standards in the sampler "in order of decreasing concentration." The standards are placed in the sampler in increasing concentration per the instrument manufacturer's specifications.

17.0 Attachments

Attachment 1: Example Data Review Checklist

18.0 Revision History

- Revision 6, dated 23 December 2010
  - Added soil matrix to Section 8.0
  - Updated Sections 7.19 and 7.20 to include soils preps.
  - Updated Sections 10.1.3 and 10.1.4.5 to include references to soil samples.

- Revision 5.2, dated 19 November 2010
  - Added reagent quality requirements to section 7.0

- Revision 5.1, dated 24 June 2010
  - Annual Technical Review.

- Revision 5, dated 24 August 2009
  - Deleted section 4.5 due to all samples, standards and rinse water use DI water not acidified solutions.
  - Deleted 0.2% sulfuric acid – not used in standard preparation
  - Added Kleenflow recipe to section 7.
  - Adjusted the Intermediate standards (CAL and ICV) to be brought to volume with DI water.
  - Added the use of Ottawa sand to the LCS and MB.
  - Changed pH check to be performed by log-in (section 10.1.1).
  - Added sections 10.3.4 – 10.3.6.
  - Fixed the Concentration calculation.
  - Updated the checklist attachment.

- Revision 4, dated 29 May 2009
  - Updated minor formatting and grammatical errors.
  - Changed the preparation time for Sodium Phenolate from monthly to weekly.
  - Changed the concentrations of the calibration standards.
  - Changed the concentration for the LCS.
  - Changed the CCV concentrations.
  - Added 2nd CCV concentration requirement for quadratic curves.
  - Added reference to corporate calibration SOP (section 10.2.4).
  - Added the performance of an MDL study for IDOC.

- Revision 3, dated 14 March 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.

- Updated method references.
- Technical review and update throughout the SOP.

- Revision 2.0 and 2.1, dated 30 August 2002 and 21 November 2006
  - For the minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 1.0, dated 15 March 2002
  - Updated standards including ability to make mixed nitrate/ammonia standards, and added details for preparing ICV standards.
  - Changed Quanterra to STL.
  - Added requirement that hypochlorite solution be made daily.
  - Added Appendices
  - Updated pollution prevention and waste sections.

Attachment 1.

Example Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Inst

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated EDLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lots/Plates reported checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Total Organic Carbon in Soil [SW 9060]

Approvals (Signature/Date):

Dave Elkin — Date: 5/3/10
Wet Chemistry Supervisor

Adam Alban — Date: 04 May 10
Health & Safety Manager / Coordinator

Karen Kuoppela — Date: 5-4-10
Quality Assurance Manager

Robert C. Hanisch — Date: 5/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes the determination of total organic carbon in soils, sludge, and sediments using the LECO C632 TOC analyzer. The LECO instrument uses 0.20 gram quantities of sample, and so results are less prone to precision problems that are typical of the trace TOC instruments that use sample aliquots in the 10-100 mg range. The method referenced for this procedure is EPA Method 9060.

1.2 The reporting limit (RL) is 0.2% carbon or 2,000 mg/kg.

2.0 Summary of Method

The sample is treated with 6N (1:1) HCL to drive off inorganic carbonates and then dried to remove moisture and acid. Organic carbon in the sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured by an infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

3.0 Definitions

Total Organic Carbon (TOC):

The carbon measured as a result of oxidation of the sample after the removal of inorganic carbon.

4.0 Interferences

4.1 Oily samples will cause erratic results. This is minimized by homogenization of the sample.

4.2 Due to the initial purging step to remove inorganic carbon, purgeable organic carbon compounds are not effectively analyzed by this procedure.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Spent crucibles must be allowed to cool to room temperature prior to disposal.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the

method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- LECO C632 Analyzer.
- Hot Plate. The temperature must be sufficient to drive off water and HCl and completely dry the samples

6.2 Supplies

- Porcelain Combustion Boats

  NOTE: Porcelain Combustion Boats are a radioactive material.
- Small Beakers
- Spoons or spatulas
- Miscellaneous volumetric glassware

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 6N Hydrochloric Acid (1:1)

Slowly and carefully and with stirring, add 500 mL of concentrated HCL to 500 mL of deionized water. Allow to cool before use.

7.2 TOC Calibration Standard (MS/SD/CCV)

Low Level: Calcium Carbonate; 12.00%C
High Level: Potassium Biphthalate, 47.05%C

7.3 TOC Initial Calibration Verification (ICV) This standard is from a different source than that of 7.2.

Low Level: Calcium Carbonate; 12.00%C
High Level: Potassium Biphthalate, 47.05%C

7.4 TOC Calibration Standard (LCS)

This standard is purchased from an outside vendor. The true value will be dependent on the lot received.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	N/A	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

A batch is a group of no greater than 20 samples excluding QC samples (LCS, MS, MSD, Method Blanks), 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.

9.3 Method Blank (MB)

One method blank (MB) must be processed with each batch. The method blank consists of a solid blank matrix 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.

Acceptance Criteria: The method blank should not contain any analyte of interest at or above the reporting limit.

Corrective Action: If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be taken in consultation with the client and must be addressed in the project narrative.

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 all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

9.4 Laboratory Control Sample (LCS)

One LCS must be processed with each 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 for TOC in soils is performed by analyzing a 0.20 g aliquot of a purchased standard.

Acceptance Criteria: The LCS recovery must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The control limits are maintained in the LIMS.

Corrective Action: If any analyte is outside established control limits, the system is out of control, and corrective action must occur. Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Samples

One MS/MSD pair must be processed for each 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) that is prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to an MS/MSD pair. 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.

The MS and MSD for the automated method are prepared by placing 0.20 g of the soil sample to be spiked into a porcelain boat and adding an identical weight of calcium carbonate. These are mixed and combusted as a sample with the weight of the soil (0.20 g) used as the sample weight in the sample table.

Acceptance Criteria: The recovery of the analyte in the MS and MSD must fall within established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference between the MS and MSD must be no greater than the established RPD limit, which is based on historical data.

Corrective Action: If the 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 re-preparation and reanalysis of the batch.

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 MS/MSD RPD limits.

9.6 Initial Calibration Verification (ICV)

The ICV standard is analyzed immediately following the ICAL. The ICV is a second-source calcium carbonate standard with a true value of 12% carbon. The analyte recovery must fall within the 90-110% range. If it is outside the acceptance limits, check the equipment and standards, correct any problems, and then recalibrate.

9.7 Continuing Calibration Verification (CCV)

The calibration is checked at the beginning of an analytical sequence (ICV), after every ten samples (CCV), and at the end of the sequence (CCV) by measuring a CCV standard.

The CCV is calcium carbonate with a true value of 12% carbon.

The CCV recovery must be within the 90-110% range. If it is outside the acceptance limits, check the equipment and standards, correct any problems, recalibrate, and rerun all samples analyzed since the last successful CCV.

9.8 Initial and Continuing Calibration Blank (ICB and CCB)

System cleanliness is checked at the beginning of an analytical sequence (ICB), after every ten samples (CCB), and at the end of the sequence (CCB) by analyzing a blank.

The ICB/CCB for the automated method is a solid sample matrix.

Results must be less than the reporting limit. If the blank result is greater than the reporting limit, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.

10.0 Procedure

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.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Samples should be homogenized, and ground to uniform consistency if necessary. Leave out any extraneous artifacts, i.e., glass shards, large twigs, leaves, etc.

10.1.2 In a small beaker, aliquot approximately 2-3 g of sample. Slowly add 6N HCl until sample is completely moistened. If sample fizzes excessively, more 6N HCl may be needed.

10.1.3 Heat samples on a hot plate until they appear dry; then dry in oven at 104°C for at least one hour.

10.2 Calibration

10.2.1 Instrument and furnace should be left on at all times. Be sure that the furnace is reheated to 1350°C before beginning analysis.

10.2.2 If the furnace has been shut down due to maintenance or a power failure, ramp the temperature up slowly to 600°C to minimize the thermal stresses on the combustion tube.

10.2.3 Turn on the compressed air to the autosampler and the oxygen to the combustion analyzer.

10.2.4 Check that the incoming oxygen pressure is 20-40 psi and the combustion pressure is <15 psi.

10.2.5 Initial Calibration: The LECO analyzer is calibrated with calcium carbonate, a solid with a true value of 12% carbon and blanks.

10.2.5.1 Analyze three blanks

10.2.5.2 Analyze a five point standard curve (0.05g, 0.010g, 0.075g, 0.15g, and 0.25g) with the 12% carbon standard.

10.2.6 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Weigh 0.20 g of dried sample into a tared porcelain weighboat. Spread the sample evenly throughout the boat. Transfer the exact sample weight into the sample table and load the boat into the autosampler.

10.3.2 When samples are loaded, use the software to begin analysis.

10.3.3 Sample results should be less than 20% carbon so that they use the calibrated low-range IR cell. Sample results of greater than 20% carbon should be reanalyzed with a smaller aliquot.

11.0 Calculations / Data Reduction

Results are reported from the instrument as a final concentration expressed as % carbon. Multiply by 10,000 to convert to mg/kg. Samples less than 0.2% are reported as ND.

11.1 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration = $\frac{\text{mg/kg or L} = C \times V \times D}{W}$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4 Control limits are stored in and accessed from LIMS.

11.5 The detection limit for this method will vary based on the results of detection limit studies performed. Samples less than the method detection limit are reported as ND. Please refer to LIMS for current reporting limit information.

11.6 First and second level data reviews are recorded on the Wet Chemistry Data Review Checklist shown in Attachment 1.

11.7 Additional calibration calculation information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then,

calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Four aliquots of the QC check sample (independent source from the calibration) 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.
- Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs (independent source from the calibration), or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produce when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acidic waste – Waste Stream F
- Porcelain Combustion Boats: Contact Radioactive Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

- Method 9060, "Total Organic Carbon", SW-846, Third Edition, 9/86.

16.0 Method Modifications:

Item	Method	Modification
1	SW 9060	Method 9060 is designed for water samples, and requires quadruplicate analysis to overcome potential precision problems. This procedure is exclusively for soil samples, and the LECO instrument is designed for soil analysis. The sample aliquots are 10-100 times larger than are practical with most other non-dispersive IR instruments, and so the precision is acceptable with single analyses.
2	SW 9060	Method 9060 requires the use of a blender to homogenize samples. Since this procedure is for soils, the samples are ground to a uniform consistency.

17.0 Attachments

Attachment 1: Example Data Review Checklist

18.0 Revision History

Revision 3.1, dated 15 May 2010

- Annual Review
- Added section 6.3

- Revision 3, dated 15 May 2009
  - Changed aliquot size from 0.25g to 0.20g throughout the SOP.
  - Changed LCS to reflect it is a purchased standard.
  - Deleted Methanol from the safety section.
  - Modified the oven temperature in section 10.1.3 to 104°C.
  - Changed section 11.5 to read samples less than the MDL are reported as ND.
  - Updated calibration section 10.2 to reflect current practices.

- Revision 2, dated 04 April 2008
  - Integration for TestAmerica and STL operations.

- Revision 1, dated 08 August 2007
  - Method was changed to be specific to the LECO C632 instrument. Sample aliquot was changed from 0.5g to 0.25g at manufacturer's recommendation.

Attachment 1.

Example Data Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

WET CHEMISTRY COVERSHEET
Revision 6 – 8/8/07

*ANALYTE:*_____ *ANALYST:*_____ *ANALYSIS DATE:*

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

1) Special instructions followed (obtained if necessary)?		Y	N/A
2) Prep sheet complete and reviewed?		Y	N/A
3) Blanks, DCSs and CCVs within limits or properly anomalized?		Y	N/A
4) DCS limits:_____ , RPD limits:_____			
5) (if applicable) MSQC limits:_____ , RPD limits:_____		N/A	
6) Client specific criteria met?	Y	N/A	
7) Chromatograms, tapes, printouts checked?		Y	N/A
8) Significant figures reported correctly?	Y	N/A	
9) Reporting limits reflect dilutions and/or limited sample volume?		Y	N/A
10) Comments, exceptions, anomalies and SOP/method variances recorded?		Y	N/A
11) Holding times met; if not, NCM form submitted, PM/PM notified and form copied and included?	Y	N/A	
12) Rework documented; original and rework data copied and included?	Y	N/A	
13) All data being reported highlighted in blue on benchsheets?		Y	N/A
14) All data (including QC) entered into LIMS and checked against the data review printout?	Y	N/A	
15) All MS/MSD data defaulted into LIMS?		Y	N/A
16) Completed dates in LIMS reflect actual prep, reprep or analysis dates?		Y	N/A
17) Run log/benchsheet and maintenance entries entered in instrument logbook?	Y	N/A	
18) Were scheduled required due dates met?		Y	N
If not, why?:_____			

*Analyst:*_____ *Date:*

1) Calculations and chromatograms checked?		Y	N/A
2) Correlation coefficient is 0.995 or greater?		Y	N/A
3) All QC (blanks, DCSs, CCVs, LCBs) within limits or properly anomalized?		Y	N/A
4) Significant figures and reporting limits correct?		Y	N/A
5) NTVs (NCM form), rework, comments and anomalies documented and included?	Y	N/A	
6) Data checked against data review printout?		Y	N/A
7) For soils, is dry-weight flag set correctly in QuantIMS (run query)?		Y	N/A
8) Project and raw data copies prepared and filed?	Y	N/A	
9) All raw data copies legible?	Y	N/A	

*Reviewed by:*_____ *Date:*

Comments:

QA/Forms/Benchst/WC/ BS0022.DOC

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Color
[EPA 110.2, SM 2120B]

Approvals (Signature/Date):

Dave Elkin — Date 5/3/10
Wet Chemistry Supervisor

Adam Alban — Date 04 May 10
Health & Safety Manager / Coordinator

Karen Kuoppala — Date 5-4-10
Quality Assurance Manager

Robert C. Hanisch — Date 5/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is used for measuring the color of water derived from naturally occurring materials, i.e., vegetable residues such as bark, roots, humus and peat materials. This method is NOT applicable to color measurement on waters containing highly colored industrial wastes.

1.2 This method is usable in the 5 to 50 unit range. Higher level samples can be analyzed by dilution of the sample.

2.0 Summary of Method

2.1 Color is measured by visual comparison of a sample to platinum-cobalt standards.

2.2 One unit of color is that produced by 1 mg/L platinum in the form of chloroplatinate ion.

3.0 Definitions

There are no terms requiring definition unique to this procedure. Refer to the Glossary of the QAM for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Even slight amounts of turbidity will interfere. Samples with turbidity may be clarified by centrifugation or filtration. If turbidity is removed, the results are reported as "true color." Otherwise, the results are reported as "apparent color."

4.2 The method is pH dependent.

4.3 Absorption of ammonia by the standards will cause an increase in color.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Balance: analytical, capable of accurately weighing to the nearest 0.0001 g. The balance accuracy is verified each day of use in accordance with SOP DV-QA-0014.

6.2 Glassware: Class A volumetric flasks and pipettes as required.

6.3 Nessler tubes, matched tall form, 50 mL capacity.

6.4 Rack for Nessler tubes.

6.5 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Chloroplatinate Stock Standard, 500 units

7.1.1 To a 1000 mL volumetric flask containing approximate 500 mL of deionized water, slowly add 100 mL concentrated hydrochloric acid. Dissolve 1.246 g of potassium chloroplatinate (K2PtCl6) and 1.0 g of cobaltous chloride monohydrate (CoCl2•H20) in this mixture and dilute to 1000 mL with deionized water.

7.1.2 Alternatively, a commercially prepared standard may be used and diluted to the appropriate concentrations.

7.2 Working Standards

7.2.1 In the 50 mL Nessler tubes, prepare the following standards:

Volume Stock Standard (7.1)	Final Volume	Color Units
0.0 mL	50 mL	0
0.5 mL	50 mL	5
1.0 mL	50 mL	10
1.5 mL	50 mL	15
2.0 mL	50 mL	20
2.5 mL	50 mL	25
3.0 mL	50 mL	30
3.5 mL	50 mL	35

4.0 mL	50 mL	40
4.5 mL	50 mL	45
5.0 mL	50 mL	50

7.3 Laboratory and commercially prepared stock standards are stored at room temperature. Laboratory prepared stock standard is assigned an expiration of one year or less, and the manufacturer's expiration for commercially prepared stock standard is observed. Working standards are assigned an expiration of no more than 6 months from preparation. Working standards are stored in the Nessler tubes tightly stoppered.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	HDPE or Glass	500 mLs	Cool 4 ± 2°C	48 Hours	40 CFR Part 136.3

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Method Blank (MB)

9.1.1 The laboratory must analyze at least one MB with each batch of 20 samples. Data produced are used to assess contamination from the laboratory environment.

Acceptance Criteria: The MB result must be less than ½ the reporting limit or less than one-tenth of the concentration measured in samples.

Corrective Action: If the method blank exceeds these levels, the problem should be investigated and all associated samples reanalyzed. If elevated blank levels are encountered and color is not detected in samples, it may be possible to report the sample results together with an NCM.

9.2 Laboratory Control Sample (LCS)

9.2.1 There is no LCS associated with this method.

9.3 Sample Duplicate

9.3.1 The laboratory must analyze at least one sample duplicate with each batch of 20 samples. The Relative Percent Difference (RPD) of the sample duplicates must be within QC limits.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Calibration

This method relies on the visual comparison of the color of a sample to the color of each of 11 standard solutions, which are prepared as described in Section 7.0.

10.2 Sample Analysis

If the samples are turbid, centrifuge until the supernatant liquid is clear. Up to one hour may be required. Alternately, samples may be filtered to remove turbidity. Samples with turbidity removed are reported as "true color" instead of "apparent color".

Fill a Nessler tube to the mark with sample and place the tube into the rack between the two standards with the most similar color values. Look vertically downward through the tube, on a white surface, to determine which standard the sample most closely resembles. The sample turbidity should be low enough that it is possible to see down the entire length of the tube to the reflective surface. Be certain to consider only sample color, not any slight darkening from any remaining sample turbidity.

Dilute any samples with more than 50 units of color and reanalyze.

11.0 Calculations / Data Reduction

Multiply the observed color by any dilution made to bring the color below 50 units.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

- There is no MDL study for color.

12.2 Demonstration of Capabilities

- Each analyst performing the method must complete a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.
- Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Contents of Nessler tubes – Aqueous Acidic (Reactivity) - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method Source: Methods for the Chemical Analysis of Water and Wastes, EPA Method 110.2, 600/4-79-020

15.2 Method 2120B: "Standard Methods for the Examination of Water and Wastewater", 20th Edition.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 110.2	The upper standard prepared is 50 units.

17.0 Attachments

Attachment 1: Example Laboratory benchsheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 2.2, dated 15 May 2010
  - Annual Review
  - Added section 6.5

- Revision 2.1, dated 15 May 2009
  - Technical review performed.
  - Updated formatting.
  - Updated method and SOP references.
  - Updated the attachments.

- Revision 1, dated 07 July 2007
  - Company name was changed from STL to TestAmerica .

Attachment 1.

Example Laboratory Bench Sheet

TestAmerica Denver

Laboratory Bench Sheet
Color (Methods 110.2, SM 2120 E)
Revision 2 – June 28, 2007

Analyst:	Date:	SOP # DEN-WC-0058 Rev. 0.1

Calibration Standard Information

Source/Verification Lot#: _____ Prep Date: _____ Made by: _____

Concentration: _____(mg/L or ug/L) Expiration Date: _____

Sample	pH	Observed Color	Dilution Factor	Turbidity Removed*?	Final Color
Blank				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	

**Note: If turbidity is removed, the results are reported as "true color". Otherwise, results are reported as "apparent color".*

Comments:_____

Attachment 2.

Example Data Review Checklist

TestAmerica Denver

Wet Chemistry Data Review Checklist
Direct Measurement Methods (pH, Conductance, etc.)

Test Name/Method #: _____ **Analysis Date** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Was the instrument properly standardized?				
2. Second-source ICV analyzed immediately after instrument standardization & recovery ± 10% of true value?				
3. ICB analyzed immediately after ICV & results < the RL?				
4. CCV analyzed after every ten samples & recovery ± 10% of true value?				
5. CCB analyzed after every CCV & all results < the RL?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs reflect required dilutions or limited sample volumes?				
2. All reported results bracketed by in control CCV results?				
3. Sample analyses done within holding time?				
4. Preparation benchsheet completed and included in package (if applicable)?				
5. Special client requirements met?				
6. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
7. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
8. Are all data being reported highlighted on the benchsheet?				
9. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank ?				
2. LCS run for batch and within QC limits?				
3. Sample DUP run at required frequency and RPD within established limits?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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Phone: 303-736-0100
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Title: Hardness by Titration [EPA 130.2 and SM 2340C]

Approvals (Signature/Date):

Dave Elkin — 5/3/10 Date
Wet Chemistry Supervisor

Karen Kuoppala — 5-4-10 Date
Quality Assurance Manager

Adam Alban — 04 May 10 Date
Health & Safety Manager / Coordinator

Robert C. Hanisch — 5/4/10 Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes by EPA method 130.2 and SM 2340C with normal to high hardness.

1.2 The lower limit of quantitation using this SOP is 5 mg/L.

2.0 Summary of Method

Ethylenediaminetetraacetic acid (EDTA) and its sodium salts form a chelated soluble complex when added to a solution containing metal cations such as calcium and magnesium. If a small amount of a dye such as Calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10, the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the calcium and magnesium ions have been complexed, the solution turns from wine red to blue, marking the end point of the titration. Magnesium ion must be present to yield a satisfactory end point, so a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer.

3.0 Definitions

Total hardness: The sum of the calcium and magnesium concentrations expressed as calcium carbonate in mg/L.

4.0 Interferences

4.1 Some metal ions interfere by causing fading or indistinct endpoints or by stoichiometric consumption of EDTA. This interference can be reduced by adding certain inhibitors before titration (See Attachment 1). The figures in the table are intended as a rough guide only. For samples with a large amount of metal interference, the hardness calculation method should be considered as an alternative.

4.2 Suspended or colloidal organic matter also may interfere with the endpoint.

4.3 Large amounts of suspended solids may interfere with the determination of the endpoint if the color of the solution can not readily be determined. These samples may require dilution prior to analysis.

4.4 This method is suitable for a wide range of concentrations, but in order to avoid large titration volumes, select a sample volume that requires less than 15 mL of EDTA titrant.

4.5 Because calcium carbonate can precipitate out of solution at high pH, the titration must be completed within 5 minutes of buffer addition to minimize the tendency toward calcium carbonate precipitation.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Nitirc 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.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Sodium Sulfide	Toxic	53 mg/kg (intraperitoneal)	Contact with acids liberates toxic gas. Toxic by inhalation, in contact with skin, and if swallowed. Causes burns in contact with skin and eyes. Material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Sodium Cyanide	Corrosive Poison	25 mg/m^3 IDLH (as CN) 5 mg/m^3 - TWA	May be fatal if inhaled, absorbed through the skin, or swallowed. May cause severe respiratory and digestive tract irritation with possible burns. May cause central nervous system effects and blood abnormalities. May cause severe eye and skin irritation, with possible burns. Contact with eyes may cause burns, chemical conjunctivitis, and corneal damage.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Balance: analytical, capable of accurately weighing to the nearest 0.0001g. The accuracy of the balance is checked each day it is used in accordance with DV-QA-0014.

6.2 Supplies

- Glassware: Class A volumetric flasks, graduated cylinders and pipettes as required.
- Mechanical pipettes
- Titration setup: Buret, Erlenmeyer flasks, stir plate and stir bar.
- Miscellaneous laboratory glassware and equipment.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

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 on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Buffer Solution

Dissolve 16.9 g of ammonium chloride (NH_4Cl) in 143 mL of concentrated ammonium hydroxide (NH_4OH). Add 1.25 g of magnesium EDTA (Ethylenediamine tetraacetic acid, magnesium salt) and dilute to 250 mL with deionized water. This solution is commercially available. Store tightly stoppered and replace after one month. Discard when 1 or 2 mL added to a sample fails to produce a pH of 10.0 ± 0.1 at the endpoint of the titration.

7.2 Calmagite Indicator

Use Calmagite powder or a commercially available Calmagite indicator solution.

7.3 1 N Ammonium Hydroxide

Dilute 35 mL of concentrated ammonium hydroxide to 500 mL with deionized water.

7.4 Inhibitor I: Sodium Cyanide

7.5 Inhibitor II: Sodium Sulfide nonahydrate.

Dissolve 5.0 g of sodium sulfide nonahydrate in 100 mL of deionized water. This solution deteriorates rapidly through air oxidation and should not be stored.

7.6 Stock Calcium Carbonate Standard, 1000 mg/L as Calcium Carbonate.

Use a commercially available source. This standard should be stored according to the manufacturer's specification. This standard expires as per the vendor specified expiration date.

7.7 EDTA Titrant, 0.02 N.

Dissolve 3.723 g of disodium EDTA dihydrate in deionized water and dilute to 1000 mL with deionized water. Standardize versus the stock calcium carbonate standard. Alternatively, a commercially available EDTA titrant may be used.

7.8 Reagent Water, $\leq$ 1 µmho-cm

7.9 Matrix Spike/Matrix Spike Duplicate

Prepare the MS/MSD by adding 10 mL of the calcium carbonate standard to 25 mL of sample. The spiking concentration is 400 mg/L.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Plastic/Glass	25 mLs	HNO_3, pH < 2; Cool 4 $\pm$ 2°C	180 Days	40 CFR Part 136.3

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Batch Definition

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 laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.2 Method Blank (MB)

One method blank (MB) must be processed with each batch. The MB consists of reagent water that is carried through the entire analytical procedure, including preparation and analysis. When analyzing soils, a soil MB is prepared using washed Ottawa sand. The MB 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 MB is prepared in the same manner as the samples using a DI water leach in accordance with SOP DV-WC-0036.

Acceptance Criteria: The MB should not contain any analyte of interest at or above the reporting limit (RL).

Corrective Action: If the analyte level in the MB exceeds the reporting limit for the test, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be taken in consultation with the client and must be addressed in the project narrative.

If the analyte concentration is greater than the reporting limit (RL) in the samples associated with an unacceptable MB, 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 all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

9.3 Laboratory Control Sample (LCS)

At least one LCS must be processed with each batch. The LCS consists of reagent water to which a known amount of target analyte has been added. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process.

Acceptance Criteria: In-house control limits, set at ± 3 standard deviations around the historical mean, are used as long as they are at least as tight as the regulatory limits. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Corrective Action: If LCS recoveries are outside established control limits, the analytical system is out of control and corrective action must be taken.

If recoveries are above control limits and sulfate is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

In other circumstances, the entire batch must be re-prepared and reanalyzed.

For hardness analysis, prepare the LCS by adding 10 mL of the calcium carbonate standard (7.9) to 25 mL of deionized water. The spiking concentration is 400 mg/L.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

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) that is prepared and analyzed along with the sample and matrix spike.

One MS/MSD pair must be processed for each batch. Some client-specific data quality objective (DQOs), may require an MS per 10 samples. This is listed in the client requirement checklist.

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 on only the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

Preparation of the MS/SD is described in Section 7.9.

Acceptance Criteria: In-house control limits, set at ± 3 standard deviations around the historical mean, are used for recovery

acceptance as long as they are at least as tight as the regulatory limits.

Corrective Action: If the calculated recovery or relative percent difference (RPD) is outside the acceptance range, the recovery of that analyte in the LCS must be within the established control limits. 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.

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Calibration and Standardization

10.1.1 Standardization of EDTA Titrant: The EDTA titrant must be standardized daily.

10.1.1.1 Place 50 mL of deionized water in a 125 mL Erlenmeyer flask.

10.1.1.2 Add 10.0 mL of stock calcium carbonate standard, 1000 mg/L (7.6)

10.1.1.3 Add 2 mL of buffer solution (7.1)

10.1.1.4 Add a small amount of powdered Calmagite or Calmagite indicator solution.

10.1.1.5 Titrate slowly with EDTA titrant (7.7), stirring continuously until the last red color disappears. The end point color is blue. Complete the titration within 5 minutes of adding the buffer solution.

10.1.1.6 Repeat and use the average of the two results as the EDTA titrant concentration.

10.2 Sample Analysis

10.2.1 Use a 25-ml sample volume, or select a volume of sample that will require less than 15 mL of EDTA titrant, but at least of 3 mL of EDTA titrant. The titration must be completed within 5 minutes of the addition of the buffer solution.

10.2.2 Place the sample aliquot into a 125-mL Erlenmeyer flask. If the aliquot used is less than 25 mL, add sufficient deionized water to bring the volume to 25 mL.

10.2.3 Add 2 mL of buffer solution.

10.2.4 If interferences are known or suspected, add one of the inhibitors at this point. For details, see Table I. If inhibitor I (sodium cyanide) must be used, be certain to do all work in a hood. Waste generated when inhibitors are used must be disposed of separately from normal waste. (See Section 15, Waste Management, for details).

10.2.5 Add a small amount of Calmagite powder or approximately 4 drops of Calmagite indicator solution.

10.2.6 Titrate with standard EDTA titrant, slowly and with continuous stirring, until the last reddish tint disappears. Solution should be blue at the endpoint. The titration must be completed within 5 minutes of the addition of the buffer solution.

NOTE: If the endpoint is not clear, interferences may be present. The sample is diluted and qualified. If a client specifically indicates that the sample contains a significant amount of heavy metals, the laboratory will use the appropriate inhibitor.

11.0 Calculations / Data Reduction

11.1 Titrant Standardization

$$\text{Normality of EDTA Titrant} = \frac{0.2}{\#\ mL\ of\ EDTA\ used\ for\ standardization}$$

11.2 Calculation of Hardness:

$$HARDNESS = \left(\frac{(VOLEDTA)(N)(50000)(DF)}{mL\ sample} \right)$$

Where:

HARDNESS	=	Hardness, mg/L as $CaCO_3$.
VOLEDTA	=	Volume of EDTA required for titration of sample, mL
N	=	Normality of EDTA
mL sample	=	mL of sample aliquotted
DF	=	Dilution factor of sample before it was aliquotted

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven standards at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in mg/L, and the standard deviation of the concentration(s) in mg/L. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P).
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows"

- Four aliquots of the QC check sample (independent source from the calibration) 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director.

DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually. Initial Demonstration of Capability

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program".

14.2 For this method, the following waste streams have been identified:

- All titration waste – Aqueous Alkaline (E)
- Expired standards and chemicals: Contact Waste Coordinator.

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated from this procedure.

15.0 References / Cross-References

15.1 Method Source: Methods for the Chemical Analysis of Water and Wastes, EPA Method 130.2

15.2 Method 2340C: "Standard Methods for the Examination of Water and Wastewater", 20th Edition, 1998.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 130.2 and SM 2340C	The reporting level for this method is 5mg/L. Therefore a lower level titration is not performed. If a lower level is required the samples will need to be analyzed by a different method (ICP).
2	EPA 130.2 and SM 2340C	The source method calls for the use of inhibitors for heavy metals. Do to the hazardous potential of the inhibitor solutions, samples will be analyzed at a dilution to minimize the affect of inhibitors unless otherwise indicated by the client.

17.0 Attachments

Appendix I: Maximum Concentration of Interferences Permissible with Various Inhibitors
Appendix II: Example Laboratory Bench Sheet
Appendix III: Example Checklist

18.0 Revision History

- Revision 3.2, dated 15 May 2010
  - Annual Review
  - Added section 6.3

- Revision 3.1, dated 15 May 2009
  - Added method modifications (section 16.0)
  - Updated method references
  - Updated formatting
  - Made minor spelling and grammatical corrections

- Changes from previous revisions
  - Section 11.4: Instruction to neutralize the sample with ammonium hydroxide was deleted.

Appendix I

Interfering Substance	Maximum Interference Concentration[a], mg/L	
	Inhibitor I	Inhibitor II
Aluminum	20	20
Barium	b	b
Cadmium	b	20
Cobalt	over 20	0.3
Copper	over 30	20
Iron	over 30	5
Lead	b	20
Manganese (Mn^{2+})	b	1
Nickel	over 20	0.3
Strontium	b	b
Zinc	b	200
Polyphosphate		10

NOTES:

a. Table concentrations are based on a 25 mL of sample volume diluted to 50 mL for analysis.

b. Titrates as hardness.

Inhibitor use:

Inhibitor I: Use 25 mg of sodium cyanide. Add sufficient buffer to achieve pH 10.0 and make sure to use this inhibitor in a hood.

Inhibitor II: Add 1mL of Inhibitor II.

Appendix II: Example Laboratory Bench Sheet

Laboratory Bench Sheet
Hardness Titration
Revision 3 - Sept 200[illegible]

TestAmerica Denver

HARDNESS BY TITRATION

Analyst:	RDAVIS	Titration Solutions		SOP Information:	Rep. / Detection Limits
Date:	5/14/2007	Solution 1:	EDTA	Number: DEN-WC-0060	MDL 0
QC Batch:	7134274	Source:	LABCHEM	Revision: 2	Rep. Limit 3
MS Run:	7134193	Lot #:	1611170	Calibration Information	
		Normality:	0.01984		
		Exp. Date:	4/30/2008	Lot # 6312-07	
				Made By: LabChem	
				Concentration:1000	
				STD# 1247-07	
				LCS True Value: 400	400.00
				Spike True Value: 200	200.00

Lot Number	Work Order	Sample Volume	Buret Start	Buret Stop		Final mL	Concentratn mg/L	Prep D.F.	Analyt. D.F.	Final Conc mg/L	% Rec.
	titrant stdz	10	0.00	10.05		10.05	997.033	1	1	997.03	0.01990
	titrant stdz	10	0.00	10.11		10.11	1002.985	1	1	1002.99	0.01978
	BLANK	25	0.00	0.00		0.00	0.000	1	1	0.00	
	LCS	25	0.00	10.44		10.44	414.289	1	1	414.29	
	LCS	25	0.00	9.99		9.99	396.432	1	1	396.43	
D7E080137-1	JWGKC	2	0.00	1.13		1.13	560.521	1	1	560.52	
D7E050135-1	JWDJQ	25	0.00	9.82		9.82	389.686	1	1	389.69	
D7E090168-1	JWJ40	10	0.00	0.50		0.50	49.604	1	1	49.60	
2	JWJ49	10	0.00	0.36		0.36	35.715	1	1	35.71	
D7E120139-1	JWVJQ	1	0.00	0.85		0.85	843.261	1	1	843.26	
2	JWVJ2	1	0.00	0.79		0.79	783.737	1	1	783.74	
3	JWVJ3	25	0.00	0.00		0.00	0.000	1	1	0.00	
4	JWVJ9	10	0.00	4.79		4.79	475.203	1	1	475.20	
5	JWVKE	0.5	0.00	0.40		0.40	793.658	1	1	793.66	
6	JWVKN	10	0.00	0.72		0.72	715.284	1	1	715.28	
7	JWVKQ	1	0.00	2.63		2.63	2609.150	1	1	2609.15	
8	JWVKV	5	0.00	3.40		3.40	674.609	1	1	674.61	
1S	JWVKV	5	0.00	13.60		13.60	2698.437	1	1	2698.44	
1SD	JWVKV	5	0.00	14.17		14.17	2811.533	1	1	2811.53	0.05882
											0.01471

Appendix III: Example Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver Wet Chemistry Data Review Checklist For Titration Methods

Test Name/Method #: _____ **Analysis Date** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Was the normality of the titrant verified and found acceptable?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs/MDLs reflect required dilutions or limited sample volume?				
2. All reported results bracketed by in control LCS or QC Sample?				
3. Sample analyses done within holding time?				
4. Initial pH check documented for all samples (if required)?				
5. Preparation benchsheet completed and included in package (if applicable)?				
6. Special client requirements met?				
7. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
8. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
9. Are all data being reported highlighted on the benchsheet?				
10. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x method blank result?				
2. LCS run for batch and within QC limits?				
3. MS and/or MSD run at required frequency and within limits (if applicable)?				
4. Sample DUP run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

Comments: _____

2nd Level Reviewer : _____ **Date:** _____

Comments: _____

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Title: Determination of solids in waters and wastes [EPA160.1, EPA160.2, EPA160.3, EPA160.4,SM 2540B, SM 2540C, SM 2540D, and SM 2540E]

Approvals (Signature/Date):

Dave Elkin — 11/1/10 — Date
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Health & Safety Manager / Coordinator

John P. Morris — 11/2/10 — Date
Quality Assurance Manager

Robert C. Hanisch — 11/1/10 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP is applicable to the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids using gravimetric techniques. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.2 The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 4 mg/L - 20,000 mg/L). As a practical matter, the final residue weight should be limited to about 200 mg.

1.3 The procedure for settleable solids is in SOP # DV-WC-0032.

1.4 The procedure for percent moisture in solid samples is in SOP # DV-WC-0023.

2.0 Summary of Method

2.1 **Total Solids (TS):** A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105 °C. The increase in weight over that of the empty dish represents the total solids.

2.2 **Total Dissolved Solids (TDS):** A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.3 **Total Suspended Solids (TSS):** A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.4 **Volatile Solids (VS):** The residue obtained from the determination of total solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solids.

2.5 **Volatile Dissolved Solids (VDS):** The residue obtained from the determination of total dissolved solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.6 **Volatile Suspended Solids (VSS):** A well-mixed sample is filtered through a glass fiber filter to separate the suspended material. The filter is dried and weighed, then ignited at 550 °C and reweighed. Volatile suspended solids is determined from the weight loss after ignition. The filter from the analysis of TSS may be used for the determination of VSS.

3.0 Definitions

3.1 **Total Solids (TS):** The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105 °C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter. The reporting limit is 10 mg/L.

3.2 **Total Dissolved Solids (TDS):** Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

3.3 **Total Suspended Solids (TSS):** Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

3.4 **Volatile Solids (VS):** The portion of total solids which is lost on ignition at 550 °C.

3.5 **Volatile Dissolved Solids (VDS):** The portion of total dissolved solids which is lost on ignition at 550 °C.

3.6 **Volatile Suspended Solids (VSS):** The portion of suspended solids which is lost on ignition at 550 °C.

3.7 **Aliquot:** A representative portion of a sample.

3.8 **Reagent Water:** Deionized water which is free of the analyte(s) of interest.

4.0 Interferences

4.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2 Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

4.4 Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

4.5 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

4.6 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7 The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8 Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10 Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11 Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.12 Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

4.13 Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.15 The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Environmental Health and Safety Manual), laboratory coat, and nitrile or latex 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.

5.2 Primary Materials Used

There are no materials used in this method that 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.

6.0 Equipment and Supplies

6.1 Analytical balance capable of weighing to 0.0001 g. The balance calibration is checked each day of use with three Class 1 weights that bracket the range of use. Details for this procedure are covered in SOP DV-QA-0014.

6.2 Vacuum filtration apparatus.

6.3 Vacuum pump equipped with moisture trap.

6.4 Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent. The laboratory currently uses pre-washed and pre-weighed filters purchased from an outside vendor. If these pre-prepared filters are not available, filters can be prepared as stated below.

6.4.1 Preparation of Glass Fiber Filter Disc for TSS/VSS

6.4.1.1 Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface up.

6.4.1.2 While vacuum is applied, wash the disc with three successive (approximately) 20 mL volumes of distilled water.

6.4.1.3 Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

6.4.1.4 Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish and dry in an oven at 103-105 °C for one hour.

6.4.1.5 Remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

6.4.1.6 Weigh the cooled filter and aluminum weighing dish to the nearest 0.1 mg using an analytical balance. Record the weight and the dish identification number on the benchsheet.

6.5 Glass beakers for TDS, minimum 150 mL volume, must be thoroughly cleaned, rinsed with de-ionized water and baked at 180 ± 2 °C for TDS and 104 ± 2 °C for TS at least one hour before use. Transfer to a desiccator and allow to cool completely before use.

Note: Glass beakers may not be used for procedures requiring a muffle furnace. In that case, porcelain dishes, pre-dried and weighed, must be used.

6.6 Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.

6.7 Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.8 Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.

6.9 Muffle furnace (550 °C ± 50 °C).

6.10 Thermometers, NIST traceable.

6.11 Conductivity meter and associated apparatus.

6.12 Graduated cylinders, assorted sizes.

6.13 Volumetric flasks, Class A, assorted sizes.

6.14 Aluminum weighing dishes large enough to hold a 47 mm filter.

6.15 Forceps for handling filters.

6.16 Crucible tongs.

6.17 Zetex gloves or other gloves capable of providing protection at 550 °C.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent (see also Section 10.1.3).

7.2 LCS solution (500 mg/L TDS):

Place 500.0 mg of sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months. Alternatively, a commercially available LCS solution may be used.

7.3 Commercially available reference materials are used for the TSS/TS LCS.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Method	Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
TS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TDS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TSS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VDS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VSS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3

9.0 Quality Control

9.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL) as described in Section 12.1.

9.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.2 for more details.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See QC Policy (DV-QA-003P) for further details.

9.4 Method Blank

A method blank is required with every batch of 20 or less samples. The blank is deionized water taken through the procedure like a sample. The general requirement is that the method blank result must be less than the reporting limit or one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL). Note that some agencies (e.g., South Carolina) require that the blank must be less than the MDL – see special requirements in LIMS to determine which criterion applies.

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be reanalyzed

9.5 Laboratory Control Sample (LCS)

One LCS is required with each analytical batch. Historical control limits are based on three standard deviations of past results, and must be 80-120% or tighter. The process of establishing control limits is described in more detail in the QC Policy DV-QA-003P.

Corrective Action: If the LCS exceeds allowable levels, all associated samples must be reanalyzed.

9.6 Duplicate Sample Analysis

Two duplicate pairs are required with each analytical batch. The relative percent difference (RPD) must be within 10%. Note that the control limits only apply to samples with results greater than 5 times the RL. The process of establishing control limits is described in more detail in the QC Policy DV-QA-003P.

Corrective Action: If the RPD is greater than 10 the sample should be reanalyzed.

10.0 Procedure

- 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 (see SOP # DV-QA-0031 for details) and must be 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 nonconformance with a cause and corrective action described.
- All samples are to be checked out of sample control with the chain of custody documentation filled out completely.
- Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.
- If possible, analyze 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.
- Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.
- If samples are visibly oily, this should be noted on the benchsheet.
- If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the benchsheet and reporting limits must be adjusted appropriately.

10.1 Calibration

10.1.1 Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by following the lab-specific balance calibration SOP. Analytical balance calibration verification must be performed daily (every 24 hours).

10.1.2 Oven temperature must be checked daily and recorded either on the benchsheet or in an oven temperature logbook.

10.1.3 Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25 °C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

10.2 Sample Preparation

10.2.1 Total Solids

10.2.1.1 If only total solids are to be measured, heat clean dish to 103-105 °C for one hour. If volatile solids are to be measured in addition to total solids, ignite the clean evaporating dish at 550 °C for one hour in a muffle furnace.

10.2.1.2 Remove the dish from the muffle furnace using tongs and heat resistant gloves.

10.2.1.3 Cool and store dish in desiccator until dish reaches room temperature or until needed.

10.2.1.4 Weigh immediately before use to the nearest 0.1 mg. Record the weight into the LIMS benchsheet.

10.2.2 Total Dissolved Solids

Preparation of beakers/evaporating dishes.

- If only total dissolved solids are to be measured, heat clean beakers to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, ignite the clean evaporating dish at 550 ± 50 °C for one hour in a muffle furnace.
- Heat resistant gloves and tongs must be used when removing items from the muffle furnace.
- Store and cool dish in desiccator until dish reaches room temperature or until needed.

Note: Analyst must transfer the dish with gloves or tongs to prevent added weight due to oil from fingerprints.

- Weigh immediately before use to the nearest 0.1 mg. Record the weight of the dish into the LIMS benchsheet.

10.2.3 Total Suspended Solids

The pre-washed and pre-weighed filters come in aluminum pans that have scan bars that are associated with an ID and weight of each filter. These IDs are scanned into the LIMS system. If filters need preparation, see Section 6.4.1.

10.3 Sample Analysis

10.3.1 Total Solids

10.3.1.1 Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of sample (to the nearest mL) on the benchsheet.

10.3.1.2 Choose an aliquot of sample sufficient to contain a residue of at least 10 mg but less than 200 mg.

10.3.1.3 If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. Prescreening should be performed using a conductivity meter to determine the required sample volume or dilution. Dilution should be the smallest dilution sufficient to bring the approximate conductivity to under 3000 umho/cm.

10.3.1.4 For the LCS, measure 100 mL of the LCS Solution (Section 7.3) and pour into the dish.

10.3.1.5 For the MB, measure 100 mL of reagent water and pour into the dish.

10.3.1.6 Place the tray of samples into a drying oven and evaporate to dryness.

10.3.1.7 Dry the evaporated sample for at least one hour at 103-105 °C.

10.3.1.8 Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.1.9 Remove the tray of weighing dishes from the oven using heat-resistant gloves. Place in a desiccator and cool to room temperature.

10.3.1.10 Weigh the dish to the nearest 0.1 mg. Record the weight on the LIMS benchsheet.

10.3.1.11 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

10.3.1.12 If volatile solids are to be determined, treat the residue according to Section 10.3.4.

10.3.1.13 Calculate results according to the equation provided in Section 11.3.1. Use the final weight achieved for calculating TS.

10.3.2 Total Dissolved Solids

10.3.2.1 Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

10.3.2.2 Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, pre-weigh the prepared filter and refer to Section 10.3.3 for additional guidance.

10.3.2.3 Transfer 100 ml or a larger volume to yield between 10 and 200 mg dried residue to the funnel by means of a graduated cylinder. If more than 10 minutes are required to complete filtration, decrease sample volume or increase filter size.

Note: Multiple filters may be used if performing only TDS analysis.

10.3.2.4 The conductance of each sample may be used to determine the appropriate sample volume to process.

Note: TDS is typically 55-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing non-ionic species or samples with conductance greater than 10,000 umho/cm or less than 50 umho/cm.

10.3.2.5 If the sample has a conductance less than 3,000 umhos/cm, 100 mL should be used.

10.3.2.6 If the conductance is > 3,000 umhos/cm, the smallest dilution that would bring the conductance to under 3,000 umhos/cm should be used.

10.3.2.7 A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record on the benchsheet the reason that a smaller volume had to be used and any sample observations.

10.3.2.8 Record the volume of sample used (to the nearest mL) on the benchsheet.

10.3.2.9 For the method blank, process 100 mL of reagent water as the sample. Blank result should be less than method detection limit.

10.3.2.10 For the LCS, process 100 mL of the LCS Solution. Refer to Section 7.2 for instructions on how to prepare the LCS.

10.3.2.11 Filter the sample through the glass fiber filter.

10.3.2.12 Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow

for complete drainage between washings. Continue to apply vacuum until the filter is completely dried.

10.3.2.13 Transfer the filtrate (including the washings) to a pre-weighed evaporating dish on a tray. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

10.3.2.14 Evaporate the samples to dryness in a drying oven.

10.3.2.15 Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

10.3.2.16 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.2.17 Use heat resistant gloves to remove the tray of samples from the oven. Place in a desiccator and cool to room temperature.

10.3.2.18 Weigh the dish to the nearest 0.1 mg. Record the combined weight of the dried residue and the dish on the LIMS benchsheet.

Note: If the sample residue is over 200 mg (0.2 g) the sample needs to be re-analyzed at a dilution or an NCM needs to be written.

10.3.2.19 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.2.20 Calculate results according to the equation in Section 12.2. Use the final weight achieved for calculating TDS.

10.3.3 Total Suspended Solids

10.3.3.1 Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters only with forceps.

10.3.3.2 Selection of Sample Volume

- For a 47 mm diameter filter, sufficient sample to yield between 10 mg and 200 mg of dried residue. Some clients or agencies (e.g. South Carolina) require increasing standard volume from 250 mL up to 1L to achieve a minimum residue of between 10 and 200 mg.

- Because it can be difficult in some samples to determine the amount of TSS present visually, record on the benchsheet all observations on samples for which the entire volume could not be filtered due to slowing of filtration.
- If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

  Note: If the sample appears high in TSS, start with a smaller sample volume.

10.3.3.3 Invert and shake the sample vigorously, then quickly aliquot the sample. It is important to pour out the sample immediately after shaking so that the solids do not have time to settle. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 10.3.2 for additional guidance.

10.3.3.4 Remove all traces of water by continuing to apply vacuum after the sample has passed through.

10.3.3.5 With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

10.3.3.6 Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

10.3.3.7 Carefully remove the filter from the filter support and transfer to an aluminum weighing dish. If the filter is torn or damaged during this process, the sample must be reanalyzed. Take care to keep the filter face-up during the transfer so that the residue does not fall off.

10.3.3.8 Dry the filter at least one hour at 103-105 °C.

10.3.3.9 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.3.10 Use heat resistant gloves to remove the tray of dishes from the oven. Place in a desiccator and cool to room temperature.

10.3.3.11 Cool the samples in a desiccator (minimum of 1 hour) weigh (to the nearest 0.1 mg), and record the weight on the benchsheet.

10.3.3.12 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.3.13 If volatile suspended solids are to be determined, treat the residue according to Section 10.3.6.

10.3.3.14 Calculate the results using the formula given in Section 11.3.2. Use the final weight achieved for calculating TSS.

10.3.4 Volatile Solids

10.3.4.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.4.2 Place evaporating dish containing residue generated by Total Solids protocol (Section 10.3.1) in muffle furnace to ignite the residue.

10.3.4.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.4.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.4.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.4.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

10.3.4.7 Return the samples to the muffle oven for another cycle repeating steps 10.3.4.2 – 10.3.4.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.4.8 Calculate the results using the formula given in Section 11.3.4. Use the lowest weight achieved for calculating VS.

10.3.5 Volatile Dissolved Solids

10.3.5.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.5.2 Place evaporating dish containing residue generated by Total Dissolved Solids protocol (Section 10.3.2) in muffle furnace to ignite the residue. The dish used should be a ceramic weighing dish, not a glass beaker.

10.3.5.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.5.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.5.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.5.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling to room temperature.

10.3.5.7 Return the samples to the oven for another cycle of weighing. Repeat steps 10.3.5.2 to 10.3.5.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.5.8 Calculate the results using the formula given in Section 11.3.5. Use the lowest weight achieved for calculating VDS.

10.3.6 Volatile Suspended Solids

10.3.6.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.6.2 Place glass fiber filter disc containing residue generated by Total Suspended Solids protocol (Section 11.12) in muffle furnace to ignite the residue.

10.3.6.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.6.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.6.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.6.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling to room temperature.

10.3.6.7 Return the samples to the oven for cycle of weighing, repeating steps 10.3.6.3 – 10.3.6.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.6.8 Calculate the results using the formula given in Section 11.3.6. Use the final weight achieved for calculating VSS.

11.0 Calculations / Data Reduction

- If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.
- All data are subject to two levels of review, which is documented on a checklist (see example Data Review Checklist in Attachment 3).
- If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

11.1 Conversion equation

All sample are weighed in grams but reported in milligrams. Use the following equation before computing further calculations:

Weight in grams X 1000 = Weight in milligrams

11.2 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.3 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.4 Concentration

11.4.1 Total Solids =

$$\text{Total Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

11.4.2 Total Dissolved Solids =

$$\text{Total Dissolved Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

11.4.3 Total Suspended Solids =

$$\text{Total Suspended Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

11.4.4 Volatile Solids =

$$\text{Volatile Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

11.4.5 Volatile Dissolved Solids = Volatile Dissolved Solids, mg/L $= \frac{(A - B) \times 1000}{C}$

Where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

11.4.6 Volatile Suspended Solids = Volatile Suspended Solids, mg/L $= \frac{(A - B) \times 1000}{C}$

Where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- The MDL studies and concentrations can be found at L:\QA\Read\MDL.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows"

- 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.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 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.

- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

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. Further details concerning the training program are described in SOP# DV-QA-0024.

13.0 Pollution Control

Prescreening is performed to determine the required sample volume or dilution in order to minimize laboratory waste. In addition, standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents to be disposed.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produce when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acidic sample waste – Waste Stream F
- Filter and filter residue – nonhazardous waste
- Solid soil waste – Waste Stream S

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated from this procedure.

15.0 References / Cross-References

15.1 Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, and 160.4.

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1992: Methods 2540A, 2540B, 2540C, 2540D, and 2540E.

16.0 Method Modifications:

None

17.0 Attachments

Attachment 1: Example TDS/TS/TSS Benchsheet

Attachment 2: Example VSS/VDS/VS Benchsheet

Attachment 3: Data Review Checklist

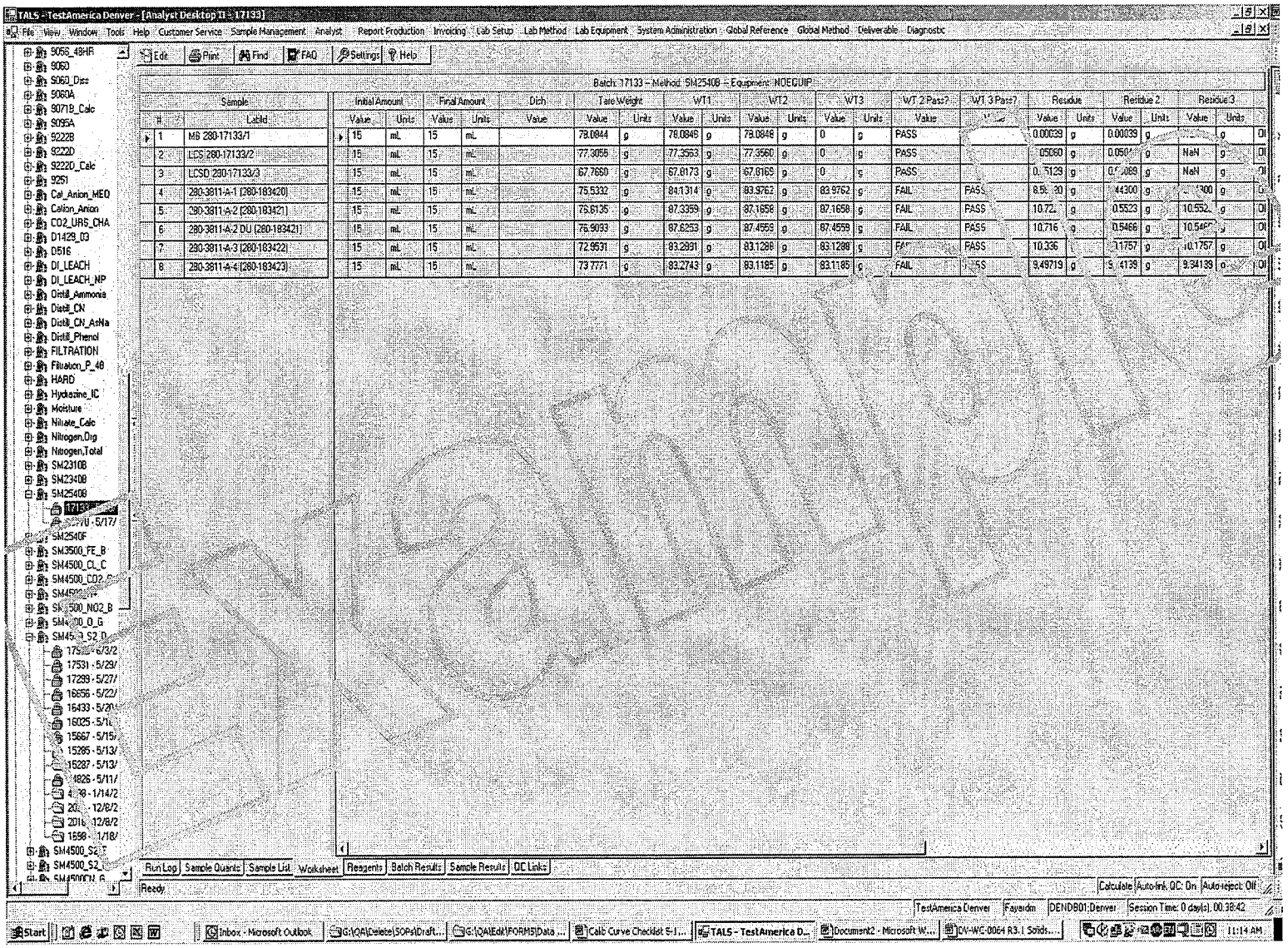
18.0 Revision History

- Revision 5.1, dated 19 November 2010
  - Corrected the duplicate acceptance criteria in section 9.6.
- Revision 5, dated 08 October 2010
  - Added Note to section 10.3.2.18 concerning sample residue weights.
- Revision 4, dated 18 June 2010
  - Annual Technical Data Review
  - Updated SOP to include the use of pre-washed & weighed filters.
  - Updated all attachments to include the new LIMS bench sheets.
- Revision 3.1, dated 19 June 2009
  - Corrected minor grammatical and formatting errors.
  - Technical and method review performed.
- Revision 3, dated 16 June 2008
  - Fixed formatting
  - Added EPA references to SOP
- Revision 2, dated 21 December 2007
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Updated to include the 10% sample duplicate requirement from Standard Methods.
- Revision 1.1, dated 30 December 2005
  - Safety and Waste section updates. The technical interim changes are maintained, and the revision was increased by "X.1"
- Revision 1, dated 26 August 2002
  - Definition for TS in 3.1 corrected to have drying temperature (103-105 degrees C) match text.
  - Standard volume for TSS and VSS changed from 250 ml to 100 ml.
  - Changed references to STL from Quanterra.
  - Updated references in Quality Control Section.
  - Added Appendices with bench sheet and data review checklist.
  - Changed volume required for TSS to be up to 1L of sample, to yield 1 mg of residue weight.
  - Added requirement of some clients for a duplicate 1 per 10 samples to 9.4.3.2.
  - Added requirement of some clients to control results down to the MDL to 9.4.1.1.
  - Added reference to the balance SOP to 6.1.
  - Changed "weight loss" to "weight difference" in description of cyclical weighings.

- Removed commercially prepared filters for TSS from the procedure.
- Update to section 13 to include information on MDLs & IDCs.
- Removal of reference to MS and MSD.

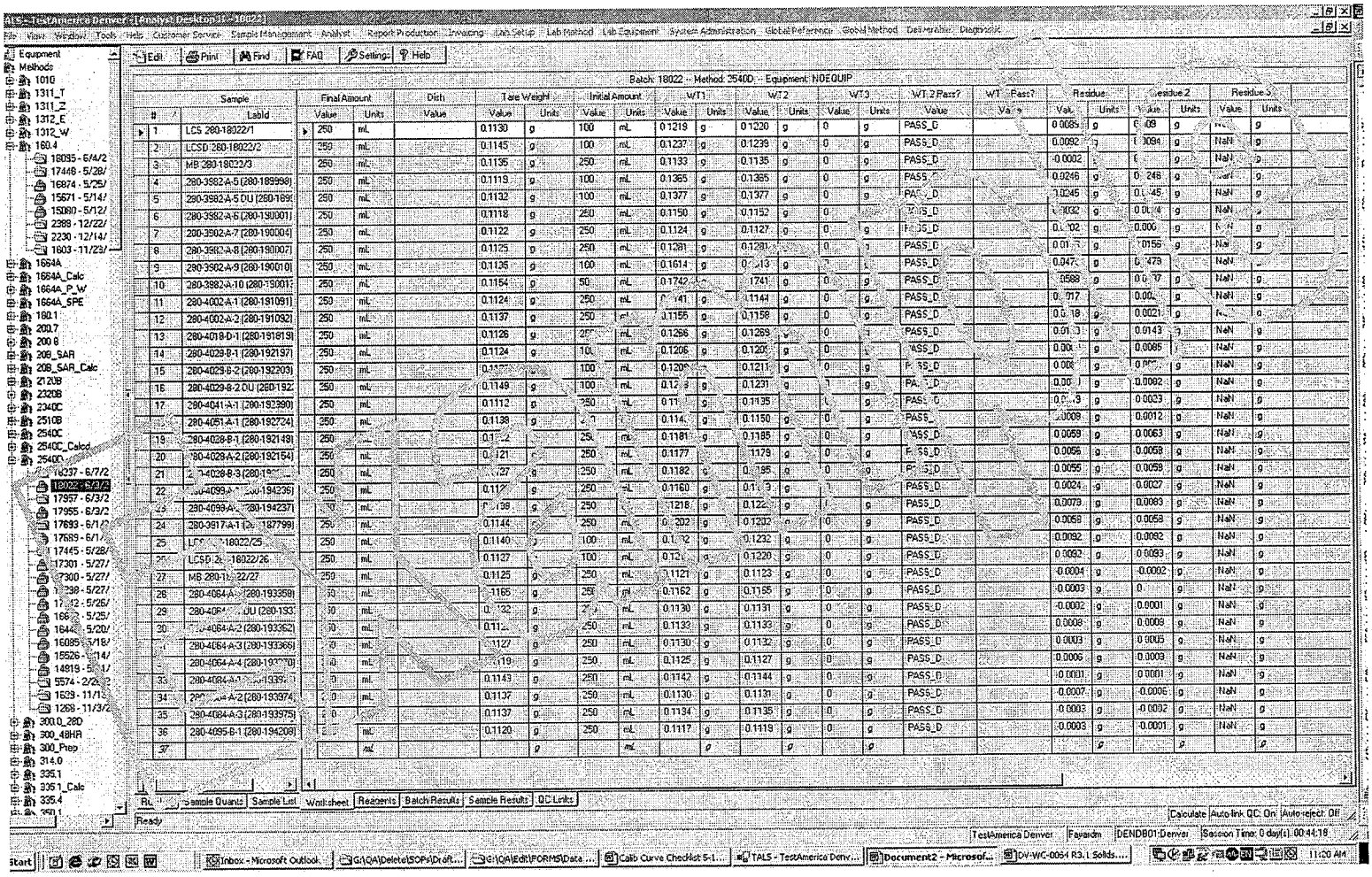
Attachment 1.

Example TDS/TS/TSS Benchsheet



Attachment 2.

Example VSS/VS/VDS Benchsheet



Attachment 3.

Data Review Checklist

TestAmerica Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist For Gravimetric Methods

Test Name/Method #: _____ **Analysis Date:** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Lot / Sample Numbers	Matrix	Prep Batch	Batch	Method	Special Inst

A. Balance, Oven, and DI Water QC Checks	Yes	No	N/A	2nd Level
1. Was the balance calibration verified before and after processing samples and noted in the "Balance Calibration Log" for the date(s) the samples were processed?				
2. Was the oven temperature within method requirements and recorded in the "Oven Temperature" logbook for the date(s) the samples were processed?				
3. Was the daily conductivity check of the deionized water recorded in the "Conductivity Logbook"?				
B. Method Requirements				
1. If sample is visibly oily, was this noted on the benchsheet?				
2. Was final residue weight within minimum/maximum requirements?				
3. Were the initial and final drying dates and times recorded on the benchsheet and were all samples dried for at least one hour?				
C. Sample Results				
1. TDS/Conductivity ratio or historical data checked?				
2. For % Moisture, was the Final Dried Weight < the Initial Pan Weight or is the result greater than 100%?				
3. Were sample analyses done within holding time?				
4. Were special client requirements met?				
5. Were data that were manually transcribed from instrument printouts into TALS verified 100% including significant figures and units?				
6. Do the prep and analysis dates in TALS reflect the actual dates? Lots/Dates report checked?				
7. STD/True Value information is updated and included?				
8. Are raw data copies prepared, scanned, and uploaded?				
D. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10 X RL?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. DUP run for batch and RPD < 20% for samples >5 X RL?				

Analyst: _____ **Date:** _____

Comments: _____

2nd Level Reviewer : _____ **Date:** _____

Comments: _____

TestAmerica
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TestAmerica
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Title: Total and Amenable Cyanide by SW-846 9010C, 9012A and 9013

Approvals (Signature/Date):

Dave Elkin — Date
Wet Chemistry Supervisor

Adam Alban — Date
Health & Safety Manager / Coordinator

Karen Kuoppala — Date
Quality Assurance Manager

Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure is for the determination of:

Analyte	CAS Number
Total Cyanide	57-12-5

1.1.2 This procedure is applicable to soils, domestic and industrial wastes and leachates.

1.1.3 This procedure detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine total and amenable cyanide as described in SW-846 9012A.

1.1.4 This procedure describes the reduced volume version of the methods and uses the same reagents and molar ratio to meet the quality control and performance requirement stated in the method.

1.1.5 Dynamic Range
The approximate working range extends from 0.01 mg/L to 0.4 mg/L (0.5 mg/kg to 20 mg/kg). Samples with higher concentrations are analyzed with dilution. Current method detection limits are given in the laboratory LIMS system.

2.0 Summary of Method

2.1 Total cyanide – Cyanide, as hydrocyanic acid (HCN) is released from samples by means of reflux-distillation under acidic condition and absorbed in a scrubber containing sodium hydroxide (NaOH) solution. The cyanide concentration in the scrubber solution is determined using an automated analyzer. The cyanide is converted to cyanogen chloride by reactions with Chloramine-T that subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color intensity which is proportionate to the cyanide concentration is measured at 570 nm.

3.0 Definitions

3.1 **Cyanide:** The term "cyanide" refers to all of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN- by the various chemical methods. These compounds include both simple and complex cyanides.

4.0 Interferences

4.1 Oxidizing agents such as chlorine will destroy cyanide. Sodium arsenite is used to remove chlorine interferences.

4.2 Samples that contain sulfide compounds may produce hydrogen sulfide during the distillation and interfere with color development. This is treated by adding an excess of bismuth nitrate to the sample prior to distillation.

4.3 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide. Chlorine present in samples from chlorinated sources will also destroy cyanide and must be destroyed before distillation.

4.4 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide. Sulfamic acid is added to remove the nitrate and/or nitrite interference.

4.5 Samples containing surfactants may foam excessively during distillation.

4.6 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation.

4.7 Amino acids may distill with the cyanide and interfere with the analysis.

4.8 Fatty acids may interfere by forming soaps in the absorption solution.

4.9 Thiocyanate greater than 10 mg/L may interfere.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, latex or nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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, and 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, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
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.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m3	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.
Potassium Hydroxide	Corrosive Poison Reactive	2 mg/m3 - ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness and swelling. Greater exposures cause severe burns with possible blindness.
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.
Sulfuric acid	Corrosive Poison Irritant Carcinogen	1mg/m3 TWA	Inhalation symptoms may include irritation of the nose and throat, and labored breathing. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, and diarrhea. Skin contact can cause redness, pain, and severe burn. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Calcium hypochlorite	Strong oxidizer	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Glacial acetic acid	Corrosive Poison Flammable Irritant	10 ppm TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Swallowing can cause severe injury leading to death. Skin contact may include redness, pain, and skin burns. Eye contact may cause severe eye damage followed by loss of sight.
Sulfamic acid	Corrosive Irritant	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Barbituric acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as a potential health hazard; do not ingest.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns, and eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Build-up of pressure in the distillation apparatus will cause the hot, acidic solution to spray out of the thistle tube. In case vacuum is lost, the condensers must be opened to prevent build-up of pressure. If the solution overflows onto the heating block, turn it off. Unplug and replace the heating mantle when it cools.

5.4 All distillations are to be performed with adequate ventilation.

5.5 Exposure to chemicals must be maintained as low as reasonable 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.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. For cyanide amenable to chlorination, the chlorination step will also be performed in a fume hood.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Denver associate. The situation must be reported immediately to a laboratory supervisor and the Health and Safety Officer.

6.0 Equipment and Supplies

6.1 Instrumentation

- Alpkem Automated Segmented Flow Analyzer
  - Autosampler
  - Proportioning pump
  - Injection module
  - Colorimeter with 570 nm filter and 10 mm flow cell
  - WinFlow Software
  - Debubblers
  - Miscellaneous tubing and reaction coils.

- Midi-distillation apparatus consisting of 100 mL distillation tubes, cold-finger condensers, absorption tubes, and associated apparatus.
- Vacuum pump.
- Recirculating chiller.

6.2 Supplies

- Disposable auto-sampler vials or culture tubes for samples.
- Eppendorf pipettes, various sizes.
- Volumetric flasks, class A, various sizes.
- Volumetric pipettes, class A, various sizes.
- Miscellaneous laboratory apparatus and glassware.

7.0 Reagents and Standards

All Standards purchased from vendors are NIST traceable. Reagents used in the lab for standard preparation must meet ACS specifications.

7.1 Cyanide Calibration Stock Standard, 1,000 mg/L

7.1.1 This standard is purchased commercially.

7.1.2 *Alternatively*, it can be made as described here.

7.1.2.1 Dissolve 2.51 g of dried (103 °C) potassium cyanide and 2.0 g potassium hydroxide in water and dilute to 1,000 mL.

7.1.2.2 This stock solution will be standardized initially and every 30 days thereafter as described below.

7.1.2.3 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the results of the weekly standardization.

7.2 Standardization of the Stock Cyanide Solution

7.2.1 Indicator: Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL acetone.

7.2.2 Silver nitrate ($AgNO_3$) titrant: 0.0192 N: Obtain a commercially prepared certified standard.

7.3 Cyanide Standardization

7.3.1 Add 10 mL of the 1000 mg/L Cyanide Stock Standard (Section 7.1) solution to a 500 mL Erlenmeyer flask. Dilute to 250 mL with deionized water.

7.3.2 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution.

7.3.3 Titrate with standard silver nitrate, $AgNO_3$, to the first change in color from a canary yellow to a salmon hue.

7.3.4 Prepare a blank in a similar fashion. Add 250 mL of deionized water to a 500 mL Erlenmeyer flask.

7.3.5 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution. Titrate with standard AgNO3 to the first change in color from a canary yellow to a salmon hue. The standardization should be done in duplicates.

7.3.6 Calculate the true stock cyanide concentration as follows:

$$\text{mg/L Stock Cyanide} = \frac{[(A - B) \times 1000]}{\text{mL of sample}}$$

A = Volume of $AgNO_3$ for titration of sample, mL

B = Volume of $AgNO_3$ for titration of blank, mL

7.3.7 If the verification result is within 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the Standards Log. A new Standards Log entry is not required.

7.3.8 If the verification result is less than 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the Standards Log.

7.3.9 Create a "new" standard in the Standard Log for this standard using the new concentration from the verification process.

7.3.10 Prepare a label for the stock solution with the name of the analyst, date of verification, expiration date (one month beyond the date of re-standardization), lot number, 1% (0.25N) NaOH, and the actual stock cyanide concentration.

7.3.11 The true concentration of the stock cyanide solution after standardization with silver nitrate is used to prepare intermediate and working standards. This concentration does not always equal 1000 mg/L. The dilution factor relative to the stock standard is provided to aid in the calculations, where:

[True Working] = [True Stock] / dilution factor

7.4 Intermediate Standard I, 50 mg/L (20x dilution from stock)

7.4.1 Pipette 5.0 mL 1,000 mg/L calibration stock standard (Section 7.1) into a 100 mL volumetric flask.

7.4.2 Dilute to volume with 1% (0.25 N) sodium hydroxide. Prepare every 7 days.

7.5 Calibration Standards

7.5.1 Working Standard, 1.0 mg/L
Pipette 2.0 mL intermediate standard I (Section 7.4) into a 100 mL volumetric flask.

7.5.2 Dilute to volume with 1% (0.25N) sodium hydroxide and mix. Prepare daily.

7.5.3 Initial Calibration Standards
Dilute the 1.0 mg/L cyanide working standard with 1% (0.25N) sodium hydroxide as follows:

Standard Level	Intermediate	Aliquot (mL)	Final Volume (mL)	Concentration (mg/L)
1	1 mg/L	10.0	25	0.40
2	1 mg/L	10.0	50	0.20
3	1 mg/L	5.0	50	0.10
4	0.1 mg/L	2.0	4.0	0.05
5	1 mg/L	1.0	50	0.02
6	1 mg/L	0.5	50	0.01
7	N/A	0	50	0.00 (Blank)

7.5.4 Calculate the exact concentration for each calibration curve standard by dividing the exact working cyanide standard concentration by the dilution factors shown above if stock concentration is different from 1.0 mg = 1.0 ml.

7.6 Continuing Calibration Verification Standard (CCV), 0.2 mg/L:
The level 2 calibration standard described in 7.5 is used as the working CCV standard.

7.7 Second-Source Standards

7.7.1 Initial Calibration Verification (ICV) Stock Standard, 1,000 mg/L: The second-source standard is obtained from a different source than the calibration standards. It is prepared and standardized as described in Section 7.2 for the primary standard. This standard is available commercially.

7.7.2 Intermediate ICV (second-source) Standard, 10 mg/L: Pipette 1.0 mL 1,000 mg/L ICV (7.7.1) stock into a 100 mL volumetric flask. Dilute to volume with 1% (0.25N) sodium hydroxide.P

7.7.3 Prepare every 7 days.Working ICV (second-source) Standard, 0.10 mg/L

7.7.4 Spike 1 mL of the 10 mg/L intermediate (7.7.2) into a 100 mL volumetric flask and fill to the mark with 1% (0.25N) NaOH.

7.8 Pyridine-Barbituric Acid Solution (per OI Manual)

7.8.1 In a hood, place 15 g barbituric ($C_4H_4N_2O_3$) acid in a 1000 mL volumetric flask and add about 100 mL deionized water, rinsing down the sides of the flask.

7.8.2 Place on a magnetic stirrer and add a stir bar.

7.8.3 Add 75 mL pyridine while mixing.

7.8.4 Carefully add 15 mL concentrated hydrochloric acid while mixing.

7.8.5 Add 500 mL of DI water and stir until the barbituric acid is dissolved.

7.8.6 Dilute to volume with deionized water and filter through a 0.45 µm filter.

7.8.7 Store in an amber bottle.

7.8.8 Expires 6 months from preparation.

7.9 Phosphate Buffer Solution 1M (per OI Manual)

Dissolve 138 g sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \cdot H_20$) in deionized water and dilute to 1000 mL.

7.9.1 Add 4 mL of Brij-35 to the solution and mix gently.

7.9.2 Store at 4°C.

7.9.3 Filter the solution through a 0.45 µm filter.

7.9.4 This solution expires 3 months from preparation.

7.10 Chloramine-T (per OI Manual)

7.10.1 Dissolve 1.0 g Chloramine-T in deionized water and dilute to 250 mL.

7.10.2 Prepare fresh daily.

7.11 Sodium Hydroxide, 10N

7.11.1 Dissolve 400 g sodium hydroxide in deionized water.

7.11.2 Cool to room temperature, dilute to 1000 mL, and mix well.

7.11.3 Store in a plastic bottle. Solution is commercially available.

7.12 Sodium Hydroxide, 2% wt/wt (0.5N)

7.12.1 Dissolve 20 g sodium hydroxide in deionized water.

7.12.2 Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.13 Sodium Hydroxide Dilution Solution, 1% wt/wt (0.25N)

7.13.1 Dissolve 10 g sodium hydroxide in deionized water.

7.13.2 Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.14 Sulfuric acid, concentrated.

7.15 Sulfuric acid, 0.02 N

7.15.1 In a 2000 mL volumetric flask, carefully add 1 mL concentrated sulfuric acid to approximately 1900 mL deionized water.

7.15.2 Dilute to final volume of 2000 mL with deionized water and mix. Solution is commercially available.

7.16 Bleach
Fragrance free commercial liquid bleach is purchased and used in place of the Calcium Hypochlorite solution.

7.17 Magnesium Chloride solution
This reagent is purchased though an approved vendor.

7.18 Acetate buffer

7.18.1 Dissolve 410 g sodium acetate trihydrate in approximately 150 mL deionized water.

7.18.2 Adjust the pH to 4.5 with glacial acetic acid and dilute to final volume of 500 mL with deionized water.

7.19 Ascorbic acid crystals.

7.20 pH test strips.

7.21 Lead acetate test paper.

7.22 Potassium iodide-starch test paper.

7.23 Boiling chips.

7.24 Sulfamic Acid, 10% wt/wt
Dissolve 10 g sulfamic acid in 100mL of deionized water. Mix well. Expires 1 year from preparation.

7.25 Brij-35 Start-Up Solution
Concentrated Brij-35 is a buffer solution obtained from the equipment vendor. The start-up solution is prepared by diluting 4 mL of the Brij-35 concentrate to 500 mL with reagent water.

7.26 Bismuth Nitrate

7.26.1 Obtain a clean, dry 100 mL volumetric flask.

7.26.2 Add approximately 20 mL of DI water to the flask.

7.26.3 Add 3 g $Bi(NO)_3 \cdot 5H_2O$ to the flask and swirl to dissolve.

7.26.4 Slowly add 25 mL of glacial acetic acid, swirling frequently.

7.26.5 Swirl until completely dissolved.

7.26.6 Bring to volume with DI water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters[1]	HDPE, Glass	500 mLs	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	40 CFR Part 136.3

[1] Add 1.2 g of ascorbic acid per liter of sample if residual chlorine is present.

9.0 Quality Control

9.1 Sample QC: The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit or < 5% of sample concentration
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4], but no more than 85-115% recovery.
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Statistical Limits [4],but no more than 75-125% recovery.
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Statistical Limits [4],but no more than 75-125% recovery.
High Distilled Standard (HDS) (0.4 mg/L)	1 in 20 or fewer samples	± 10% of true value
Low Distilled Standard (LDS) (0.10 mg/L)	1 in 20 or fewer samples	± 10% of true value

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2 Method blank: Add 50 mL of 1% (0.25N) NaOH into a distillation flask immediately prior to distillation. For solid samples, weigh 1.0 g of Ottawa Sand into a distillation flask and add 50 mL 1% (0.25N) NaOH.

Corrective Action: The corrective action for method blank failures is re-distillation and reanalysis of all samples in the batch. If there is insufficient sample for reanalysis, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager.

9.3 LCS: Spike 5 mL of the 1.0 mg/L intermediate standard (7.5.1) into a distillation flask filled with 50 mL 1% (0.25N) NaOH. For solid samples, weigh 1.0 g of Ottawa Sand into a distillation flask and add 50 mL 1% (0.25N) NaOH.

Corrective Action: If the LCS fails, redistill and reanalyze all samples in the batch. If reanalysis is not possible, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager. See the TestAmerica Denver Policy, Quality Assurance Program, DV-QA-003P for additional guidance.

9.4 MS/MSD: Measure 50 mL of sample into a distillation flask. Spike the aliquot with 5 mL of the 1.0 mg/L cyanide standard (7.5.1). The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

Corrective Action: If MS/MSD recoveries exceed LIMS historical limits for total cyanide, the Method of Standard Additions may be employed to analyze all samples associated with a particular QC batch. Consult your Supervisor, a Technical Specialist, QA Manager, or if contractually required, the client before proceeding. Because MS/MSD results may not have a direct bearing on other samples in the batch, the appropriate corrective action is generally governed by specific project requirements. At a minimum, QC failures will be noted as anomaly and discussed in the final report.

9.5 High and Low Distilled Standards (HDS & LDS), 0.4 mg/L and 0.10 mg/L
Standards are distilled to monitor the efficiency of the distillation process. See section 7.5 for preparation instructions.

Corrective Action: Distilled standard failure results in re-distillation and reanalysis of all associated samples. One possible exception is the situation in which recoveries are greater than 110% and cyanide was not detected in the samples. In that case, a Nonconformance Memo should be prepared and the failure noted in the report together with the sample results without taking other corrective action.

9.6 Instrument QC

9.6.1 Initial Calibration Verification (ICV)

9.6.1.1 Immediately after the initial calibration, the calibration is verified using a second-source ICV standard and an initial calibration blank ICB (1% NaOH).

9.6.1.2 The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

9.6.2 Continuing Calibration Checks (CCV / CCB)

9.6.2.1 A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run.

9.6.2.2 Standard checks (ICV/CCV) must be within 10% of the expected value.

9.6.2.3 Blanks must be less than the reporting limit.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

10.0 Procedure

10.1 Sample Preparation

10.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, and chemistry, sample size, or other parameters.

10.1.2 Any variation in procedure shall be completely documented using a Nonconformance Memo (see SOP# DV-QA-0033) and is approved by a Technical Specialist and QA Manager.

10.1.3 Depending on the severity of the change and prior arrangements, the client shall be notified.

10.1.4 Check aqueous samples for sulfide prior to distillation using lead acetate paper.

10.1.4.1 Moisten the paper with 2 or 3 drops of acetate buffer, and then place 1 drop of sample on the paper.

10.1.4.2 A dark color indicates a positive test for sulfide.

10.1.4.3 Record the result as "positive" or "negative" on the bench sheet.

10.1.5 Samples that test positive for sulfide by lead acetate test paper must be set aside and distilled in a separate analytical batch (QC lot).

10.1.5.1 The entire QC lot (including quality control samples and a calibration curve containing a minimum of 3 standards) is treated for sulfide interference with bismuth nitrate (7.26) according to the method of standard addition.

10.2 Cyanide Amenable To Chlorination Sample Preparation

10.2.1 Two sample aliquots are required for the determination of cyanide amenable to chlorination. The first aliquot is distilled for total cyanide (see Section 10.3), the second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination.

10.2.1.1 Check the pH of samples with pH test strips. Record the results on the bench sheet.

10.2.1.2 Measure the sample aliquots to be chlorinated (including all quality control samples) into 100 mL beakers.

10.2.1.3 For water samples, use 50 mL of sample.

10.2.1.4 For soil samples, use 1.0 g of sample and add 50 mL 1% (0.25N) NaOH.

10.2.1.5 Clearly label the samples with the proper identification and "chlorinated" as appropriate.

Note: The chlorination process must be performed in a fume hood.

10.2.1.6 Adjust the pH of the samples in the beakers to between 11 and 12 with the 10 N Sodium hydroxide solution.

10.2.1.7 Test the samples with KI-Starch paper and add bleach solution drop-wise to sample while mixing until a positive test is obtained. A positive test is indicated by a blue or black color on the paper.

10.2.1.8 Maintain the excess chlorine level in the sample for 1 hour while keeping the pH of the samples between 11 and 12 with constant mixing (use a magnetic stirrer). Add bleach solution and sodium hydroxide as necessary.

10.2.1.9 After 1 hour, add 0.1 g portions of ascorbic acid crystals until a negative test is obtained with KI-Starch paper. Add an additional 0.1 g of ascorbic acid crystals to the sample to ensure an excess of the reagent

10.2.1.10 Transfer the contents of the beakers into distillation flasks quantitatively, rinsing with deionized water.

10.2.1.11 Proceed to section 10.3 for the distillation process.

10.3 Total Cyanide Sample Preparation

10.3.1 Check the pH of aqueous samples with pH test strips. Record the results on the bench sheet.

10.3.2 Check aqueous samples for oxidizing agents such as chlorine.

10.3.2.1 Place one drop of sample on a strip of potassium iodide (KI)-starch test paper.

10.3.2.2 A blue color indicates the need for treatment.

10.3.2.3 Record the result as "positive" or "negative" on the benchsheet.

10.3.2.4 If a positive test is obtained, add a few crystals at a time of ascorbic acid until a drop of sample produces no color on the indicator paper.

10.3.2.5 Then add an additional 0.1 g of ascorbic acid in excess.

10.3.3 Measure sample aliquots into the distillation flasks as follows:

- For water samples use 50 mL of sample.
- For solid samples use 1.0 g of sample and add 50 mL 1% (0.25N) NaOH.

10.3.4 Place 25 mL 2% sodium hydroxide into the absorption tubes.

10.3.5 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

10.3.6 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.3.7 Adjust the vacuum to provide a flow rate of approximately 2-3 bubbles per second (i.e., this is approximately 1/8-1/4 inch of foam in the scrubber) in the distillation flask.

10.3.8 Verify that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.3.9 If the test for sulfide is positive, add 5 mL of 0.062 M bismuth nitrate solution (7.26) through the thistle tube to every sample and QC sample in the analytical batch.

10.3.9.1 Samples testing positive for sulfide are distilled and analyzed as a separate analytical batch.

10.3.9.2 The samples are analyzed using the method of standard addition utilizing a 2-point spike and calculating the sample concentration using an EXCEL spreadsheet application.

10.3.10 Allow to mix for 3 minutes.

10.3.11 Add 2 mL of 10% sulfamic acid solution (7.24) through the thistle tube. Allow to mix for 3 minutes.

10.3.12 Slowly and carefully, add 2.5 mL concentrated sulfuric acid through the thistle tube.

10.3.13 Rinse the tube with a little deionized water and allow to mix for 3 minutes.

10.3.14 Add 2 mL of magnesium chloride solution (7.17) and mix. If excessive foaming is observed, add additional magnesium chloride.

10.3.15 Turn on the controller and heat the samples to boiling.

10.3.16 While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.3.17 Allow samples to reflux for 1.5 hours.

10.3.18 After 1.5 hours of refluxing, allow the samples to cool for 15 minutes while air is flowing.

10.3.19 Remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with deionized water.

10.3.20 Remove the flask and rinse their contents down the drain with running water. Residue not removed by this method must be scrubbed out.

10.3.21 Dilute the sodium hydroxide in the absorption tube to 50 mL with deionized water and store in plastic vials.

10.3.22 Place each batch of distillates in a box and store the samples at 4°C until they are analyzed.

10.3.23 At the end of the day, turn off the hood, vacuum, and chiller.

10.3.24 Proceed to Section 10.4 for colorimetric analysis of the distillates.

10.4 Instrument Set-Up & Calibration

10.4.1 Instrument Stabilization (per Alpkem, OI Company, manual)

10.4.1.1 Connect the reagent pump tubes to a reagent bottle containing the start-up solution (7.25). Start the pump, allowing the start-up solution to flow through the entire system.

10.4.1.2 Make sure that the flow cell of the detector is purged of all bubbles and the flow is stable and free from surging.

10.4.1.3 Once a stable flow is achieved, connect the reagent pump tubes to their respective reagent bottles, as shown in the schematic in Appendix IV. Allow the reagents to flow through the entire system, then, once again, verify that the flow cell of the detector is purged of all bubbles.

10.4.2 Initial Calibration

10.4.2.1 Calibration is performed daily or each time the instrument is set up using the standards shown in Section 7.5.

10.4.2.2 A minimum of five standards and a blank are required for the calibration (the high standard may be dropped if needed and sample dilutions performed appropriately).

Note: If sulfide was detected during the sample preparation step and the samples are logged for 9010B/9012A, then bismuth nitrate must be used to precipitate the sulfide. A method of additions spike must be prepared, and all calibration standards must be treated and distilled in the same manner as the samples. A minimum of five standards and a blank shall be distilled. Use the same calibration levels as shown in the table above, Section 7.5.

10.4.2.3 The calibration function is calculated by least-squares linear regression, and the correlation coefficient must be > 0.995 and the absolute value of the intercept must be less than ± 1/2 the reporting limit.

Corrective Action: If the correlation coefficient is < 0.995 or the absolute value of the intercept is too large, locate

and correct the problem and re-calibrate the instrument.

Note: If the standard curve for samples with sulfide (distilled in same manner as the samples) is not acceptable, the QC batch must be reprepared. If the second preparation does not give an acceptable calibration curve, the samples may be reanalyzed on an undistilled calibration curve with a discussion in the final report narrative.

10.4.2.4 The peak height of the synchronization (sync) standard should be > 150,000.

Corrective Action: If the peak height is < 150,000, the flow cell of the instrument must be cleaned (consult manufacturer's instructions), and then the instrument must be recalibrated

10.4.2.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.4.2.6 Initial Calibration Checks: Immediately after the initial calibration, the calibration is verified using a second-source, initial calibration verification (ICV, see preparation in Section 7.7) standard and an initial calibration blank (ICB, 1% NaOH). The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

10.4.2.7 Continuing Calibration Checks: A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run. Standard checks (ICV/CCV) must be within 10% of the expected value. Blanks must be less than the reporting limit.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≥0.995 correlation coefficient
Initial Cal. Verification (ICV)	Immediately after the calibration	± 10% of the expected value
Initial Cal. Blank (ICB)	Immediately after the calibration	Less than the reporting limit
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	± 10% of the expected value

Cont. Cal. Blank (CCB)	Prior to / after every 10 injections	Less than the reporting limit
Matrix Spike & Matrix Spike Duplicate (MS/MSD)	1 in 10 or fewer samples	≤ 20% RPD

10.5 Sample Analysis

Following instrument set up and calibration, the sample distillates are analyzed in exactly the same manner as the calibration standards. The routine run sequence is as follows:

Cal 0.00 ppm
Cal 0.01 ppm
Cal 0.02 ppm
Cal 0.05 ppm
Cal 0.10 ppm
Cal 0.20 ppm
Cal 0.40 ppm
Second-source ICV
ICB
High concentration distilled standard
Low concentration distilled standard
LCS / LCSD
Method blank
6 samples (may include MS/SD)
CCV
CCB
10 samples (may include MS/SD)
CCV
CCB
Additional cycles of 10 samples with CCV/CCB
Closing CCV
Closing CCB

10.6 Instrument Shut-Down

10.6.1 Disconnect the reagent lines and put all of them into the DI water, except the buffer line.

10.6.2 The Buffer line should be put into the bridge.

10.6.3 Rinse all tubes for at least 5 min.

10.6.4 Rinse with Kleenflow Base for 5 min.

10.6.5 Rinse tubes with appropriate rinses for 5 min.

10.6.6 Turn off instrument and pump.

10.6.7 Raise platens.

10.6.8 Empty Waste

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS/ HDS, LDS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Total Cyanide Calculation:

All routine calculations for total cyanide are performed by the instrument data system, provided dilutions and other information have been correctly entered.

11.4 Cyanide Amenable to Chlorination:

Amenable Cyanide = Total CN Unchlorinated Result – Treated Result

If the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, a corrective action and/or a discussion in the final report is required. Iron-cyanides can cause this to occur. Weak acid dissociable cyanide would be a better method for these types of samples.

11.5 Reporting Final Results:

Final results are routinely reported in mg/L for aqueous samples and in mg/kg for solid samples. Results can also be reported as ug/L or ug/kg if there are special project instructions requiring it.

Note: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.6 All data are subject to two levels of review, which is documented on the checklist shown in Attachment 2.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

12.1.1 Prepare seven samples at three to five times the estimated MDL concentration.

12.1.2 Digest and analyze the MDL standards as described in Section 10.

12.1.3 Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte.

Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.

12.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS. See Policy # DV-QA-005P for further details concerning MDL studies.

12.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities:

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample (independent source from the calibration) 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 standard.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

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 Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

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, has the required experience, and has successfully analyzed initial demonstration samples (see SOP # DV-QA-0024 for details).

13.0 Pollution Control

13.1 In general, the quantity of chemicals purchased by Test America Denver is based on expected usage during its shelf life. The volume of reagents and standards prepared for this procedure reflects anticipated usage.

13.2 Bismuth Nitrate is substituted for cadmium carbonate in the procedure.

13.3 Source reduction is achieved through the use of micro-distillation followed by an automated colorimetric determination.

13.4 The volume of hazardous waste is minimized through proper segregation and management of the various waste streams generated by this procedure

14.0 Waste Management

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 Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

14.2 The following waste streams have been identified for this method:

- Cyanide standardization waste – Aqueous Alkaline (E)
- Distilled sample – Aqueous Acidic (F)
- Distillate – Aqueous Alkaline (E)
- Alpkem process waste – Aqueous Alkaline, contains pyridine (E)
- Contents of sampler cups – Aqueous Alkaline (E)
- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

- Method 9010C, Total and Amenable Cyanide: Distillation, Revision 3, August 2002.
- Method 9012B, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 2, August 2002.
- Method 9013, Cyanide Extraction Procedure for Solids and Oils, Revision 0, July 1992.

16.0 Method Modifications:

Item	Method	Modification
1	SW 9012A	Method 9012A states the amenable cyanide test must be performed under amber light. This SOP method is performed under normal laboratory conditions.
2	SW 9012A	The reflux time for Cyanide Amenable to Chlorination and Total Cyanide has been changed to 1.5 hours versus 1.0 hours to accommodate the reflux time for samples requiring distillation under the Clean Water Act (EPA Method 335.4).

Item	Method	Modification
3	SW 9012A	Calibration is verified with an independently prepared check standard (ICV) with every analytical run and a CCV is run after every 10 samples, instead of for every 15 samples.
4	SW 9012A	There are differences among the referenced methods concerning the sodium hydroxide concentration in working standards: Method 9012A states in Section 7.4.1 that calibration standards are prepared using 50 mL of 1.25N sodium hydroxide and diluting to 250 ml, which produces a 0.25N sodium hydroxide concentration. To be sure that the standards are stable, the working standards in this SOP are prepared in 0.25N.
5	SW 9012A	The Cyanide stock standard is verified upon receipt and monthly thereafter. The standard is generally depleted long before the average expiration date of 6 months. The monthly verifications are sufficiently frequent to monitor the concentration.

17.0 Attachments

Attachment 1: Cyanide Preparation Example Bench Sheet
Attachment 2: Data Review Checklist
Attachment 3: Cyanide Distillation Apparatus
Attachment 4: Alpkem Manifold Schematic

18.0 Revision History

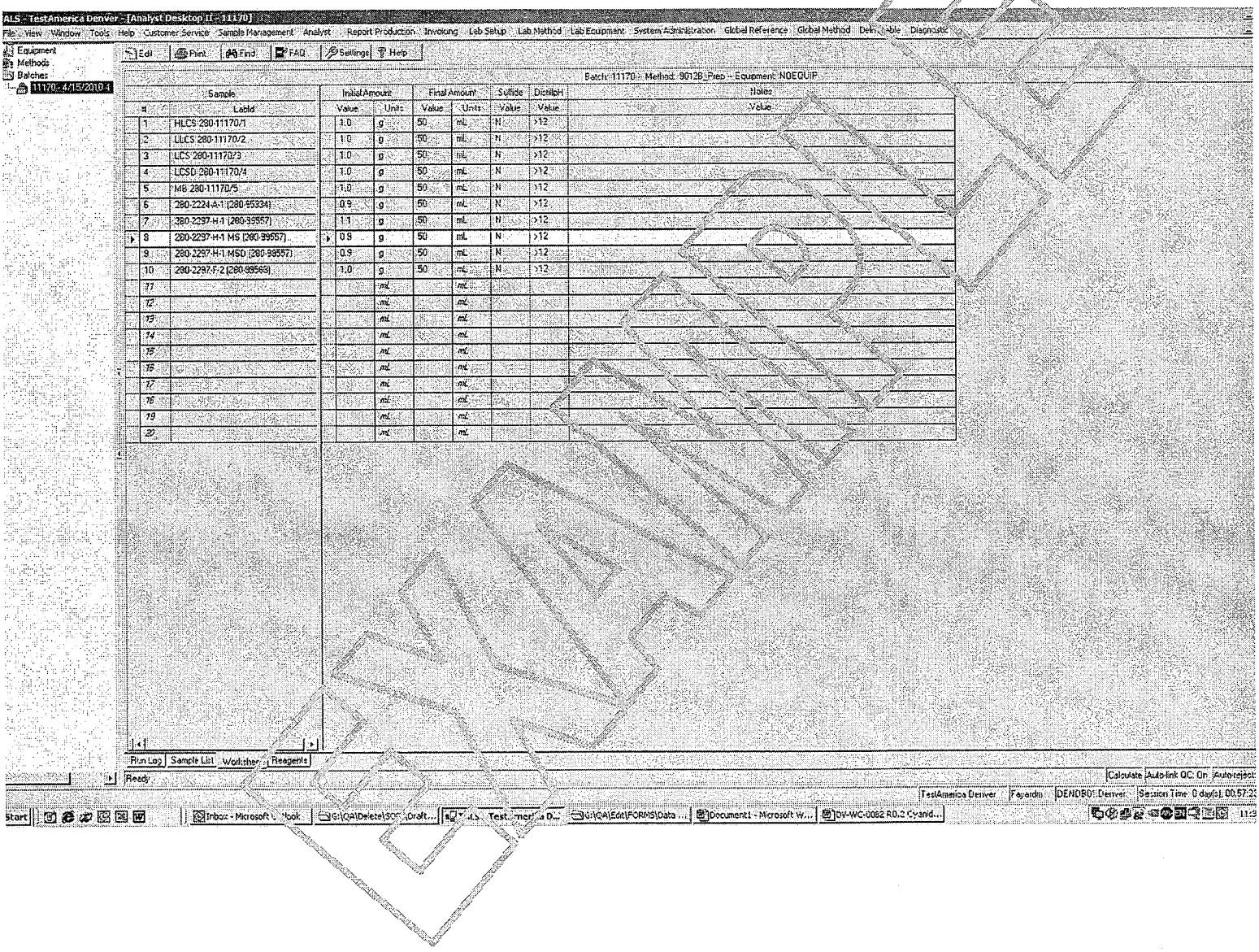
- Revision 0.2, dated 11 June 2010,
  - Annual Technical Review.
  - Updated Attachments 1 & 2.

- Revision 0.1, dated 12 June 2009, updated to changed calibration criteria to require that the intercept be less than the absolute value of the reporting limit.

- Revision 0, dated 27 February 2009

Attachment 1.

Cyanide Preparation Example Bench Sheet



Attachment 2.

Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Prep Batch	Batch	Method	Special List

	Yes	No	N/A	2nd Level
. Calibration/Instrument Run QC				
. Minimum of five standards in ICAL as specified in method?				
. Correlation coefficient ≥ 0.995 ?				
. Second-source ICV analyzed, and recovery within acceptance limits?				
. ICB analyzed immediately after the ICV & results < the RL				
. CCV analyzed after every ten samples & recovery within acceptance limits?				
. CCB analyzed after every CCV & results < RL?				
. Absolute value of the intercept is < ½ the RL?				
. Sample Results				
. All samples greater than highest calibration standard diluted and reanalyzed?				
. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
. All reported results bracketed by in control CCV results?				
. Sample analyses done within holding time?				
. Initial pH check documented for all samples? (If Applicable)				
. Preparation benchsheet completed and included in package?				
. Client requirements reviewed and met?				
. Were data manually transcribed from instrument printouts into TALS verified 100% including significant figures and correct units? (If Applicable)				
. Do the prep and analysis dates in TALS reflect the actual dates?				
1. Raw data copies prepared, scanned, and uploaded?				
2. Manual integrations done properly and initialed and dated?				
3. STD/True Value information is updated and included?				
. Preparation/Matrix QC				
. Method blank < RL or all reported samples > 10x blank have NCM?				
. Method blank < ½ RL or NCM provided?				
. LCS/LCSD run for batch and within QC limits?				
. MS/MSD run at required frequency and within limits or NCM written?				
. DUP run at required frequency and RPD within acceptance limits or NCM written?				

Analyst: _____ **Date:** _____

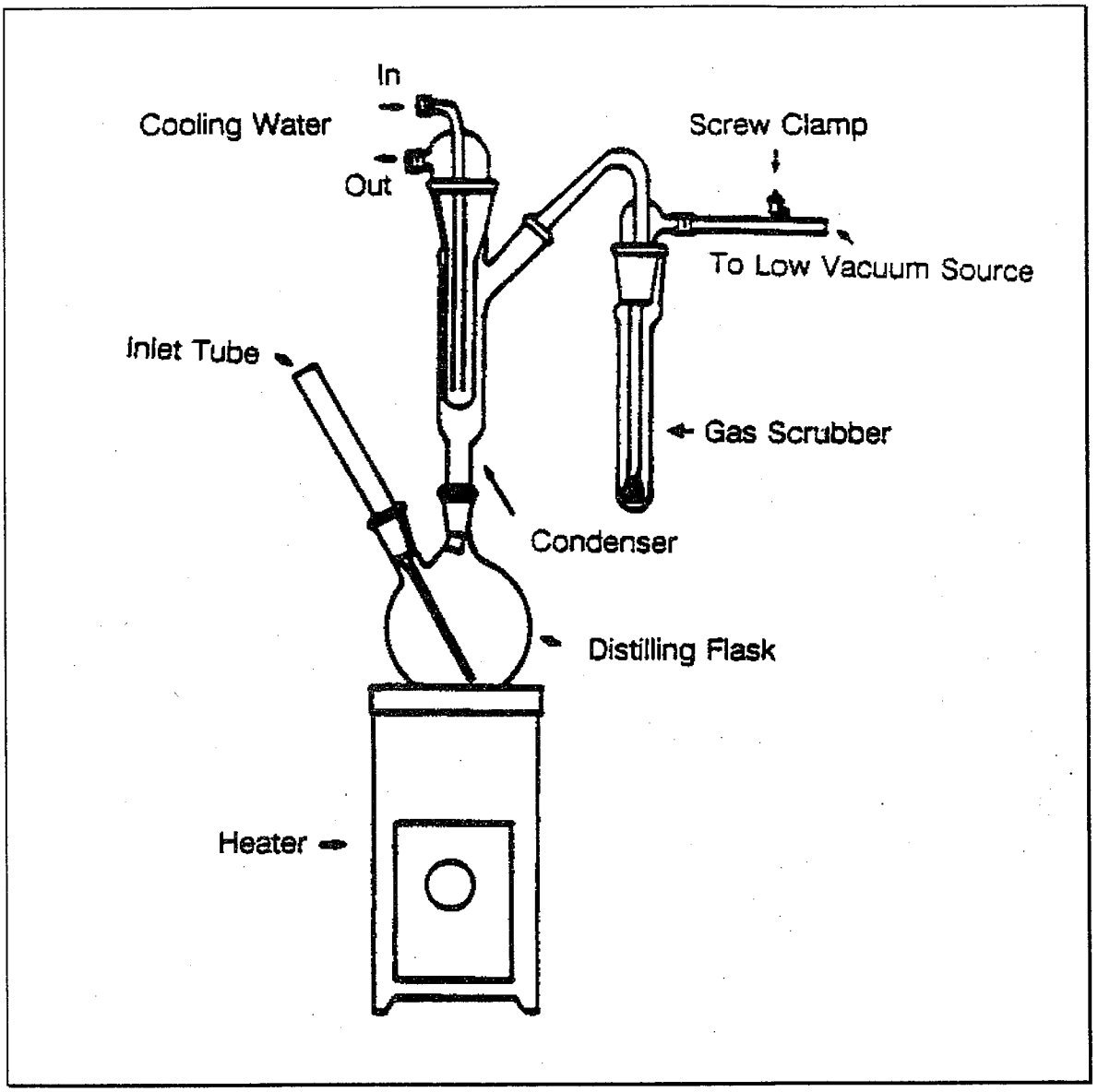
_____

_____

2nd Level Reviewer : _____ **Date:** _____

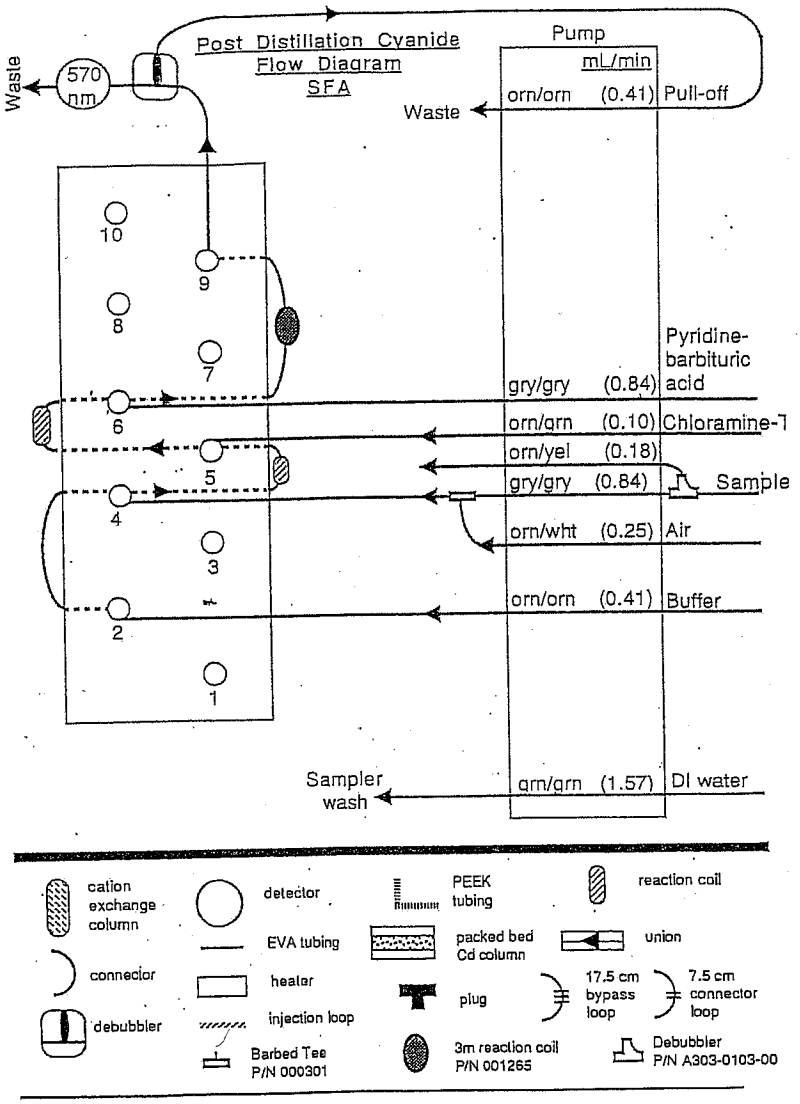
Attachment 3.

Cyanide Distillation Apparatus



Attachment 4.

Alpkem Manifold Schematic



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Title: Total Phenols, Automated Methods [EPA 420.2, EPA 420.4, SW 9066]

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of total phenols that can be distilled and subsequently measured by the 4-aminoantipyrine (4-AAP) colorimetric method.

1.2 This procedure is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.

1.3 The method determines phenol, ortho- and meta- substituted phenols, and under proper pH conditions, those para- substituted phenols in which the substitution is a carboxyl halogen, methoxyl, or sulfonic acid group. It does not determine those para- substituted phenols where the substitution is an alkyl, aryl, nitro, benzoyl, nitroso, or aldehyde group. The method does not differentiate between different types of phenol.

1.4 The routine working range for aqueous samples is 0.02 mg/L to 0.5 mg/L for the automated analysis and 0.005 mg/L to 0.1 mg/L for the chloroform extraction (manual) method. The routine working range for soil samples is 2.0 mg/kg to 50 mg/kg for the automated analysis and 0.5 mg/kg to 10 mg/kg for the chloroform extraction (manual) method. The working range can be extended by dilution of the distilled sample.

2.0 Summary of Method

2.1 The sample is acidified and distilled to separate phenolics from nonvolatile interfering compounds. A measured volume of an aqueous sample is distilled. A weighed aliquot of a solid sample is placed into a measured volume of water for the distillation. Phenolic compounds in the distillate react with 4-aminoantipyrene (4-AAP) in the presence of alkaline potassium ferricyanide to form a reddish-brown antipyrine dye.

2.2 The antipyrine dye formed by the reaction between the steam-distillable phenols and the 4-AAP is kept in the aqueous solution and the absorbance is measured at 505 nm.

3.0 Definitions

3.1 Total Phenols: The hydroxy derivatives of benzene and its condensed nuclei.

3.2 Ortho-substituted: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the substituent atoms or radicals are located in adjacent positions on the benzene ring. This is also called the 1,2 position. The ortho position on a phenol molecule is adjacent to the hydroxyl group.

3.3 Meta-substituted Phenols: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the second substituent atom or radical is located on the third carbon atom with respect to the first substituent. This is also called the 1,3 position. The meta position on a phenol molecule is separated from the hydroxyl group position by one carbon.

3.4 Para-substituted: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the substituent atoms or radicals are located in opposite positions on the benzene ring. This is also called the 1,4 position. The para position on a phenol molecule is opposite the hydroxyl group.

4.0 Interferences

4.1 Color response of phenolic materials with 4-aminoantipyrine is not the same for all phenolic compounds. Because phenolic-type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this

reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

4.2 Most chemical interferences are eliminated by distillation of an acidified sample. However, some phenolic compounds are not steam-distillable and are not included in this analysis.

4.3 Many interferences from sulfur compounds (such as sulfide, thiosulfate, and sulfite from certain industrial treatment samples), phenol-decomposing bacteria, oxidizing and reducing substances, and alkaline pH can be eliminated by acidification and aerating briefly by stirring. Sulfite interferes but is not eliminated by this treatment.

4.4 Oxidizing agents such as chlorine will oxidize phenolic compounds and are removed by the addition of excess ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

4.5 Oils can distill and interfere with the analysis. The water phase can be separated and analyzed separately. The oil phase may have to be analyzed by other methods. In any case, the client should be consulted.

4.6 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

4.7 Background contamination from plastic tubing and sample containers is eliminated by filling the wash receptacle by siphon and use of glass tubes for the samples and standards.

4.8 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The use of separatory funnels to extract aqueous samples with chloroform creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

5.1.3 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.1.4 This method uses strong oxidizers, which can cause severe burns and tissue destruction.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phenol	Corrosive	5 ppm (TWA)	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Phosphoric Acid	Corrosive	1 mg/m^3 (TWA)	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Sever 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.
Ammonium Hydroxide	Corrosive Poison	50 ppm (TWA)	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Potassium Ferricyanide	Irritant	None	**This material will form hydrogen cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. However it does not break down into cyanide compounds in the body.** May cause irritation to the respiratory tract, skin and eyes

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Balance, analytical, capable of measuring to ± 0.0001 g. The accuracy of the balance is checked each day before use in accordance with SOP DV-QA-0014.
- Recirculating chiller.
- pH meter and electrode, with automatic temperature correction
- Alpkem Flow Solution IV autoanalyzer, which includes sampler, pump, colorimeter, 505 nm filter, 10 mm flowcell, and software.

6.2 Supplies

- All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller, and associated adapters and hardware. Kontes midi distillation system is used to distill reduced volume for the modified procedure to the method.
- Eppendorf Pipettes, varying volumes
- Volumetric (Class A) glassware: varying volumes.
- Boiling stones.
- pH test strips
- Starch/iodide test strips

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent water (ASTM type II or equivalent), distilled or deionized water, free of the analytes of interest.

7.2 Sulfuric Acid, 50%:

Very slowly add 500 mL of concentrated sulfuric acid to 500 mL of deionized water with constant mixing, and allow the solution to cool completely before handling.

CAUTION: The reaction is very exothermic and should be done with extreme caution.

7.3 Sulfuric Acid, 1.0 N

This solution may be obtained from commercial sources. If prepared in the laboratory, slowly, carefully, and with stirring, add 2.78 mL of concentrated sulfuric acid to approximately 80 mL of deionized water. Dilute to 100 mL with deionized water. Allow to cool before use.

7.4 Phosphoric Acid Solution, 8.5%

Carefully add 10 mL of 85% phosphoric acid to approximately 80 mL of deionized water and bring to a final volume of 100 mL with deionized water.

NOTE: This solution is used ONLY if samples have been preserved with phosphoric acid, and the pH needs to be adjusted. Otherwise, this solution is not routinely used as part of this SOP.

7.5 Ferrous Ammonium Sulfate Solution

Add 1 mL of concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g of ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.6 Sodium Hydroxide, 2.5N

Dissolve 10 g of sodium hydroxide in approximately 80 mL of deionized water. Cool and dilute to 100 mL. Store in a plastic bottle.

7.7 Sodium Hydroxide, 1N

Dissolve 40 g of sodium hydroxide in approximately 800 mL of deionized water, cool, and dilute to 1000 mL with deionized water. Store in a plastic bottle.

7.8 Potassium Ferricyanide

Dissolve 1.0 g of potassium ferricyanide, 1.6 g of boric acid, and 1.88 g of potassium chloride in 400 mL of deionized water. Adjust the pH to 10.3 with 1 N sodium hydroxide, and dilute to 500 ml in a volumetric flask. Add 1.0 mL of Dowfax 2A1 and mix the solution gently. Prepare weekly.

7.9 4-Aminoantipyrine

Dissolve 0.065 g of 4-aminoantipyrine in approximately 80 mL of deionized water and dilute to 100 mL. Add 1.0 mL of Dowfax 2A1 and mix gently. Prepare daily.

7.10 Sampler Wash

Place deionized water under a strong vacuum for 15-20 minutes before use. Water may also be degassed with a stream of helium gas through a glass frit for approximately 5 minutes.

7.11 Dowfax Start-up Solution

To approximately 400 mL of deionized water, add approximately 1 mL of Dowfax 2A1. Dilute to 500 mL and mix gently.

7.12 Methyl Orange Indicator Solution

Dissolve 0.5 g of Methyl Orange in approximately 800 mL of deionized water. Dilute to 1 L with deionized water. This solution is also available commercially.

7.13 Phenol Stock Calibration Standard, 1000 mg/L

A commercially available standard is used.

7.14 Phenol Intermediate Calibration Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L Stock Calibration Standard (Section 7.13) to 100 mL with deionized water.

7.15 Working Calibration Standards

Dilute the 10 mg/L "automated" Intermediate Calibration Standard (Section 7.14) with deionized water as follows:

Automated Method Calibration Levels

Level	Volume of Intermediate Standard (mL)	Final Volume (mL)	Conc (mg/L)
1	2.0 of Level 2 standard	4.0	0.01

2	0.2	100	0.02
3	2.0 of Level 4 standard	4.0	0.05
4	1.0	100	0.1
5	2.0	100	0.2
6	5.0	100	0.5

NOTE: The standards are not distilled.

7.16 Phenol ICV Stock Standard, 1000 mg/L

This solution must be prepared using a standard obtained from a source different from the one that supplied the phenol calibration standard (Section 7.13). Dissolve 1.000 g of phenol in deionized water and dilute to 1000 mL. A commercially available standard can be used.

7.17 Phenol ICV Intermediate Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L ICV Stock Standard (Section 7.16) to 100 mL with deionized water.

7.18 ICV Standard

Dilute 1 mLs of the 10 mg/L ICV Intermediate Calibration Standard (Section 7.17) to 100 mL with deionized water. The true value is 0.1 mg/L.

7.19 Continuing Calibration Verification (CCV) Standard

Prepare the CCV in the same manner as the 0.2 mg/L calibration standard, as described in Section 7.15 As with the calibration standards, the CCV is not distilled.

7.20 Initial and Continuing Calibration Blank (ICB and CCB)

The ICB/CCB for the automated method is reagent water.

7.21 Phenol LCS Intermediate Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L Stock Standard (Section 7.14) to 100 mL with deionized water.

7.22 LCS Standard

Dilute 1 mLs of the 10 mg/L Intermediate Calibration Standard (Section 7.21) to 50 mL with deionized water. The true value is 0.2 mg/L. This standard is then distilled and analyzed in the same manner as a sample.

7.23 MS/MSD Standard

Dilute 1 mLs of 10 mg/L Intermediate Calibration Standard (Section 7.14) to 50 mL with the selected sample. The true value is 0.2 mg/L. These spiked samples are then distilled and analyzed in the same manner as all other samples.

7.24 Method Blank

One method blank (MB) must be processed with each 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.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	HDPE	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples. See section 7 for standard preparation.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	90-110% for EPA 420.4
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Statistical Limits [3]
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Statistical Limits [3]

Note: If all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC - The following quality control samples are prepared with each batch of samples (ICAL standards are prepared as listed). See section 7 for standard preparation.

Step	Standards	Type	Control Limit	Frequency
Initial Cal	6 Calibration standards. See section 7.18.1 for concentrations.	Linear	≥0.995 correlation coefficient	Initially or when continuing calibration verification fails acceptance criteria.
ICV/LCS	Initial calibration verification	Second source See section 7.21.1	90-110%	Initially or immediately after the ICAL.
ICB	Initial calibration blank	Reagent water	Less than RL	After the ICV.
CCV	Continuing Calibration Verification	See section 7.22.1	90-110%	Every 10 or fewer samples and after the last sample.
CCB	Continuing Calibration blank	Reagent water	Less than RL	After every CCV.
Linear Calibration Range	blank and three standards	Three calibration standards analyzed with analytical run	±10% of standards true value	Every six months

10.0 Procedure

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 an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Measure and record the pH of each water sample. pH test strips may be used.

10.1.2 Samples from chlorinated sources must be checked for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet and generate an NCM anomaly for all positive results.

10.1.3 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

10.1.4 Distillation for Automated Method

10.1.4.1 For the MIDI distillation setup, measure 50 mL of sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 0.5 g and add 50 mL of deionized water. Record the exact weight on the bench sheet.

10.1.4.2 For all soil/waste samples, add 50% sulfuric acid drop-wise until the pH is < 4 and document it on the prep sheet.

10.1.4.3 Assemble the distillation apparatus, turn on the chiller water, and start the distillation. The distillate is captured in the receiving tube of the distillation rack, which has a 50 mL volumetric mark.

10.1.4.4 When 40 mL of distillate have been collected, turn off the heating mantle and allow to cool.

10.1.4.5 Add 5 to 10 mL of water to the distillation flask and resume distillation until nearly 50 mL have been collected.

10.1.4.6 Bring the distillate to a final volume of 50 mL with deionized water.

10.1.4.7 Turn off the heating mantle and clean out the flask when cool. Do not over-distill the samples as this will lead to interferences in the analysis.

10.1.4.8 Rinse condensers with deionized water several times between samples to ensure no contamination between samples.

10.1.4.9 At the end of the day, turn off the chiller.

10.1.4.10 Refrigerate distillates at 4 ± 2 °C until they are analyzed. Analysis should follow as soon as possible.

NOTE: If the distillate is turbid, filter through pre-washed membrane filter.

10.2 Calibration

10.2.1 Automated Method Initial Calibration (ICAL)

10.2.1.1 The ICAL is performed automatically by the instrument at the beginning of each analytical sequence.

10.2.1.2 Load the standards listed in Section 7.15 into the autosampler so that they are analyzed before any samples. The calibration standards are not distilled.

10.2.1.3 The instrument software processes the calibration data and generates a calibration function that is used to calculate sample

results. The instrument software uses a least squares linear regression to relate the concentration of phenol in each standard and the associated absorbance reading, as follows:

$$y = bx + a$$ **Equation 1**

Where:

y = Absorbance of standard at 505 nm.
x = Phenol concentration of standard, mg/L.
b = Slope of the fitted straight line.
a = Y-intercept of the fitted straight line.

10.2.1.4 The instrument software uses the calibration function to calculate the concentration of each sample that is analyzed. The instrument software prints a calibration report for review, which includes the calibration equation and correlation coefficient. The correlation coefficient of the calibration line must be ≥ 0.995. If this cannot be achieved, then check the calibration standards, correct any problems, and repeat the ICAL.

10.2.1.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Automated Analysis

10.3.1.1 Place all lines in the Dowfax startup solution and place the wash lines into degassed deionized sampler wash water. Allow the system to flow for at least 10 minutes. Ensure that no leaks are present and that the flowcell of the detector is purged of all bubbles. Flow should be stable and free from surging. Ensure that the 505 nm interference filter is in place, and be sure that the lines from the autosampler and to the waste are moved to the appropriate waste container if another analysis was being performed on the instrument.

10.3.1.2 Place the reagents online and allow the system to pump for 10 to 15 minutes. Monitor the baseline at 505 nm and ensure that the baseline noise is low and that the baseline is not drifting up or down. If the baseline is drifting up or down, purge the flowcell of air bubbles before continuing.

10.3.1.3 Set up the sample table in the instrument software and load the standards and the sample and QC sample distillates into the autosampler in the order dictated by the sample table. If any sample is diluted, then make sure to enter the correct dilution factor into the instrument software. When loading, take care to ensure that the sample poured into each cup position is verified against the sample table to ensure accuracy. Following is a typical analytical sequence:

ICAL standards
ICV
ICB
Method Blank
LCS
7 samples (can include the MS and/or MSD)
CCV
CCB
10 samples (can include the MS and/or MSD)
CCV
CCB

10.3.1.4 Start the analytical sequence. Monitor the run carefully to ensure that QC and continuing calibration verification results meet the established acceptance criteria (see Sections 9). Any sample that displays a dip or a split peak should have its pH checked and adjusted to neutral before analysis.

10.3.1.5 Any sample that has a concentration that exceeds the highest standard must be diluted and reanalyzed. Samples analyzed directly after a highly concentrated sample may be adversely affected by carryover.

10.3.1.6 After the analytical sequence is done, flush the system thoroughly with deionized water and turn off the instrument. Be sure to release the pressure on the pump tubes.

10.3.1.7 Clean all glassware, apparatus and the work area.

11.0 Calculations / Data Reduction

11.1 Calculations

11.1.1 All required calculations are performed by the auto analyzer software, provided that dilutions and other information have been correctly entered.

11.1.2 The concentration in an unknown sample distillate is calculated by the instrument software by solving the calibration equation (Equation 1) for concentration (x) and using the measured absorbance of the sample, as follows:

$$x = \frac{y - a}{b}$$ **Equation 3**

Where:

x = Phenol concentration in sample distillate (mg/L).
y = Absorbance of the sample distillate at 505 nm, corrected for the blank.
b = Slope of the fitted calibration line.

a = Y-intercept of the fitted calibration line.

11.1.3 If a sample distillate was diluted, then the instrument software calculates a final result for an aqueous sample as follows (assuming that the analyst entered the correct dilution factor into the instrument software program):

$$C_s = x \times DF \qquad \textbf{Equation 4}$$

Where:

C_s = Phenol concentration in the original sample (mg/L).
x = Phenol concentration in sample distillate at the instrument (mg/L).
DF = Dilution factor.

11.1.4 For soil samples, the phenol concentration in the original sample is calculated as follows:

$$C_s = x \times \frac{V_d}{M_s} \times DF \qquad \textbf{Equation 5}$$

Where:

C_s = Phenol concentration in the original sample (mg/kg).
x = Phenol concentration in sample distillate at the instrument (mg/L).
V_d = Volume of distillate (L). For the automated method, this is usually 0.050 L (50 mL).
M_s = Mass of original sample aliquot (kg). For the automated method, this is typically 0.0005 kg (0.5 g).
DF = Dilution factor, if applicable.

11.1.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

11.2 Reporting

11.2.1 Reporting units are mg/L for water samples and mg/kg for soil samples.

11.2.2 The reporting limit for the automated method is 0.02 mg/L (2 mg/kg).

11.2.3 The reporting limit for the manual method is 0.005 mg/L (0.5 mg/kg).

11.2.4 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.2.5 All associated data are entered into the LIMS as required.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal

laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

- For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.
- Four aliquots of the QC check sample (independent source from the calibration) are analyzed with the same procedures used to analyze samples, including sample preparation.
- The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.
- 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 successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents, in this SOP, are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention," of the Corporate Environmental Health and Safety Manual, and DV-HS-001, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Distilled client sample – Aqueous Alkaline - Waste Stream E
- Contents of autosampler cups - Aqueous Alkaline - Waste Stream E
- Chloroform extracted sample waste - Aqueous Alkaline – Waste Stream E
- Chloroform extract waste – Waste Stream C

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

- Method 9066, Phenolics (Colorimetric, Automated 4-AAP With Distillation), Revision 0, September 1986.

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition; Clesceri, L.S.; Greenberg, A.E.; Eaton, A.D.; Editors; American Public Health Association, American Water Works Association, and Water Environment Federation, 1998.

15.3 Method 5530, Phenols

15.4 EPA Method 420.2, Phenolics (Colorimeteric, Automated 4-AAP With Distillation), Approved for NPDES, 1974.

15.5 EPA Method 420.4, Determination of Total Recoverable Phenolics by Semi-Automated Colorimetry, Approved for NPDES, 2007

16.0 Method Modifications:

Item	Method	Modification
1	SW 9066	There is a discrepancy between the preservation methods and holding times given in the SW-846 methods and those given in Chapter Two of SW-846. The laboratory has chosen to use sulfuric acid to adjust the sample pH to 2.
2	SW 9066, EPA 420.2, and EPA 420.4	The size of the distillation apparatus and volumes of sample and reagent were reduced to conserve space and speed up the analysis. Reduced volume versions of this method that use the same reagents

Item	Method	Modification
		and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method. It is not possible to use this method to differentiate between different types of phenols.
3	SW 9066, EPA 420.2, and EPA 420.4	Provisions have been made for dilution of the chloroform extracts up to 5x. This is sometimes necessary for samples that are over-range and cannot be reprepared due to limited sample volume or other reasons.
4	SW 9066, EPA 420.2, and EPA 420.4	Standard Methods Method 5530B distills samples by adjusting the pH to "approximately" 4.0 with H_3PO_4, while SW-846 methods distill by adjusting to a pH "<" 4 with H_2SO_4 (Method 9066) and to a pH of "approximately" 4 with H_2SO4 (Method 9065). This SOP distills the samples by adjusting the pH to less than 4 with H_2SO_4.
5	SW 9066, EPA 420.2, and EPA 420.4	The source methods state to place phenol standards in sampler "in order of decreasing concentration." The standards are placed in the sampler in increasing concentration per the instrument manufacturer's specifications.

17.0 Attachments

Attachment 1: Example Wet Chemistry Prep Bench Sheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 0, dated 28 March 2010
  - Annual Review
  - Added section 6.3

- Revision 0, dated 15 March 2009

Attachment 1.

Example Wet Chemistry Prep Bench Sheet

TestAmerica Denver

PHENOL SAMPLE PREPARATION

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

	ICV Information	ICAL/CCV/LCS/MS
Analyst: ______	Source: ______	Source: ______
	Verification/Lot #: ______	Verification/Lot #: ______
Date: ______	Prep Date: ______	Prep Date: ______
	Made By: ______	Made By: ______
Prep Time: ______	Concentration: ______	Concentration: ______
	Expiration Date: ______	Expiration Date: ______
Batch #: ______		

SOP Information	
SOP #:	DEN-WC-0013
Rev #:	

Lot Number	Work Order #	pH	Initial Vol (mL)*	Default Final Vol (mL)*	Prep Dil.	Comments
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
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			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		

** When performing the manual method circle the 200mL aliquot. When performing the automated method circle the 50mL aliquot. If a different final volume is used, cross out "50 or 200", write in correct volume, and explain in Comments column.*

Attachment 2.

Example Data Review Checklist

TESTAMERICA Denver

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery +/- 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Special client requirements met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 20x blank have NCM?				
2. LCS run for batch and within QC limits ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

2/11/09 Version
QA\Edit\Forms\Wet Chemistry\Calib Curve Checklist

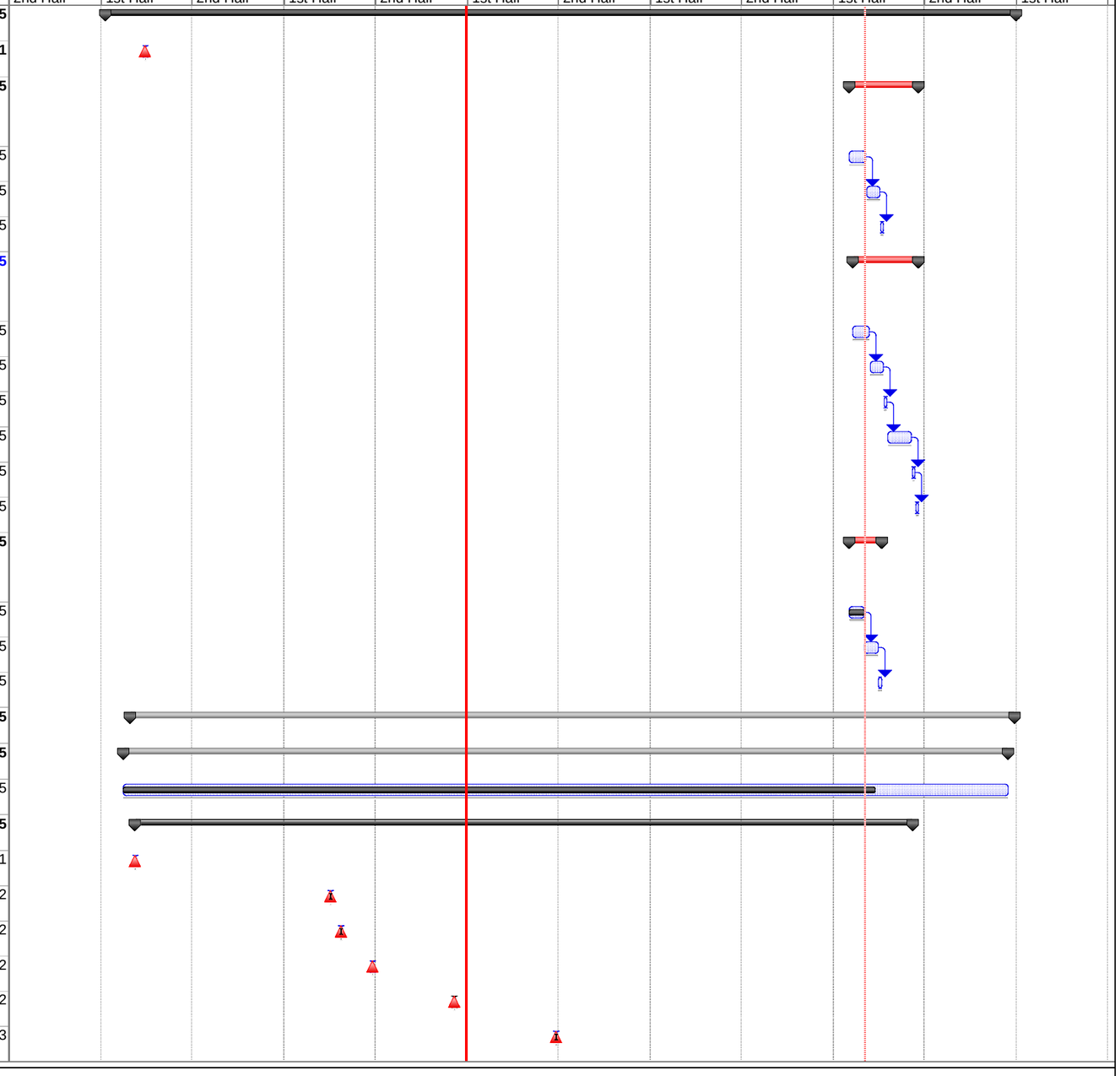
Appendix C

APPENDIX C

PROJECT SCHEDULE

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York
FA8903-10-D-8595 Delivery Order 0014
Updated 3/6/15

ID	Task Name	Duration	Start	Finish
1	**Griffiss AFB IMS Project Duration POP**	**1299 days**	**Mon 1/10/11**	**Thu 12/31/15**
2	**Kick Off Meeting at Griffiss AFB**	**2 days**	**Wed 3/30/11**	**Thu 3/31/11**
3	**Final PMP Revision (2010, 2011, 2012, 2013, and 2014 revisions completed).**	**100 days**	**Mon 2/2/15**	**Fri 6/19/15**
4	PMP Updates Draft 2015	25 days	Mon 2/2/15	Fri 3/6/15
5	Air Force Review of Draft 2015 PMP Update	20 days	Mon 3/9/15	Fri 4/3/15
6	Final 2015 PMP Update	5 days	Mon 4/6/15	Fri 4/10/15
7	**Final UFP QAPP Revision (2010, 2011, 2012, 2013, and 2014 revisions completed).**	**95 days**	**Mon 2/9/15**	**Fri 6/19/15**
8	2015 Draft UFP-QAPP Update	25 days	Mon 2/9/15	Fri 3/13/15
9	AFCEC Review 2015 Draft UFP-QAPP Update	20 days	Mon 3/16/15	Fri 4/10/15
10	Preparation of Draft Final 2015 UFP-QAPP	5 days	Mon 4/13/15	Fri 4/17/15
11	AFCEC and Regulatory Review of 2015 UFP QAPP Update	35 days	Mon 4/20/15	Fri 6/5/15
12	Preparation of Final 2015 UFP-QAPP	5 days	Mon 6/8/15	Fri 6/12/15
13	Submittal of Final 2015 UFP-QAPP Update	5 days	Mon 6/15/15	Fri 6/19/15
14	**Final SSHP Revisions (2010, 2011,2012, 2013, and 2014 revisions completed).**	**47 days**	**Mon 2/2/15**	**Tue 4/7/15**
15	2015 Draft SSHP Update	22 days	Mon 2/2/15	Tue 3/3/15
16	Air Force Review of Draft 2015 SSHP Update	20 days	Wed 3/4/15	Tue 3/31/15
17	Final 2015 SSHP Update	5 days	Wed 4/1/15	Tue 4/7/15
18	**Monthly Progress Meetings**	**1261 days**	**Mon 2/28/11**	**Mon 12/28/15**
20	**CPSMR Monthly Progress Reports**	**1261 days**	**Tue 2/15/11**	**Tue 12/15/15**
21	CPSMR Monthly Progress Reports	1261 days	Tue 2/15/11	Tue 12/15/15
22		**1108 days**	**Thu 3/10/11**	**Mon 6/8/15**
23	Semiannual Meeting 1	2 days	Thu 3/10/11	Fri 3/11/11
24	Semiannual Meeting 2	1 day	Tue 4/3/12	Tue 4/3/12
25	Semiannual Meeting 3	1 day	Tue 4/24/12	Tue 4/24/12
26	Semiannual Meeting 4	1 day	Tue 6/26/12	Tue 6/26/12
27	Semiannual Meeting 5	1 day	Thu 12/6/12	Thu 12/6/12
28	Semiannual Meeting 6	1 day	Thu 6/27/13	Thu 6/27/13



Bars with red highlights are completed tasks. Blue or black bars are active sites and associated active tasks.

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York
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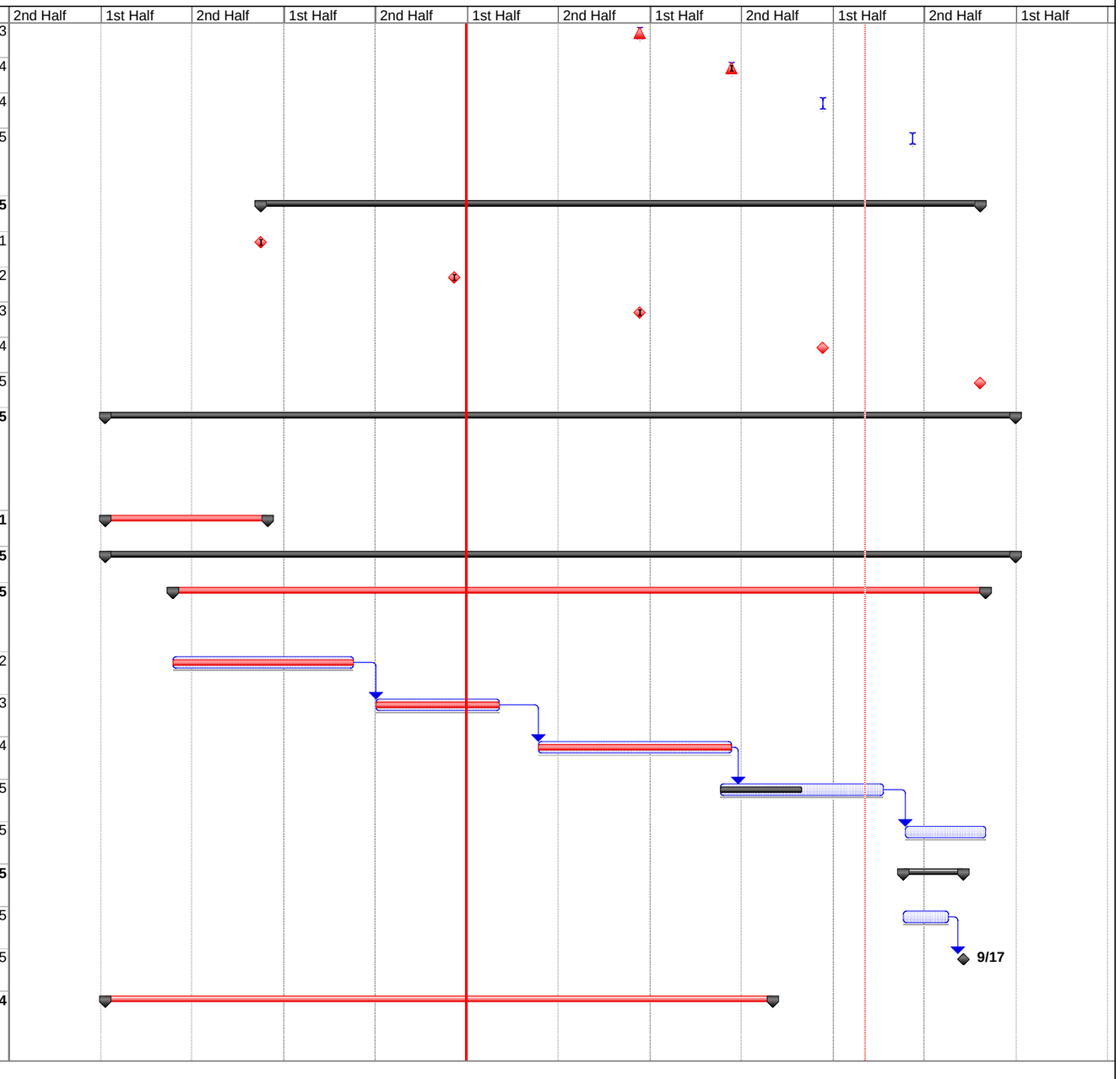
ID	Task Name	Duration	Start	Finish
29	Semiannual Meeting 7	1 day	Wed 12/11/13	Wed 12/11/13
30	Semiannual Meeting 8	1 day	Thu 6/12/14	Thu 6/12/14
31	Semiannual Meeting 9 (Postponed due to Winter Storm Damon)	1 day	Thu 12/11/14	Thu 12/11/14
32	Semiannual Meeting 10 (Combine with Meeting 9 and move up to early May 2015?)	1 day	Mon 6/8/15	Mon 6/8/15
33	**RAB/BCT Meeting**	**1026 days**	**Wed 11/16/11**	**Wed 10/21/15**
34	RAB/BCT Meeting 1	1 day	Wed 11/16/11	Wed 11/16/11
35	RAB/BCT Meeting 2	1 day	Thu 12/6/12	Thu 12/6/12
36	RAB/BCT Meeting 3	1 day	Wed 12/11/13	Wed 12/11/13
37	RAB/BCT Meeting 4 (Postponed. Reschedule Early May 2015)	1 day	Thu 12/11/14	Thu 12/11/14
38	RAB/BCT Meeting 5	1 day	Wed 10/21/15	Wed 10/21/15
39	**Landfill Sites (LF001 - Landfill 1, LF002-Landfill 2/3, LF003-Landfill 7, LF007-Landfill 5, and LF009-Landfill 6) Optimized Exit Strategy**	**1298 days**	**Mon 1/10/11**	**Thu 12/31/15**
40	**LF - Planning Documents (Completed)**	**232 days**	**Mon 1/10/11**	**Wed 11/30/11**
43	**LF - Field Work (Completed)**	**1298 days**	**Mon 1/10/11**	**Thu 12/31/15**
49	**LF - Reporting (Annual Reports completed for 2011, 2012, and 2013)**	**1158 days**	**Tue 5/24/11**	**Sun 11/1/15**
50	LF - Annual Monitoring Report - Year 1-2011	360 edays	Tue 5/24/11	Fri 5/18/12
51	LF - Annual Monitoring Report - Year 2-2012	246 edays	Tue 7/3/12	Wed 3/6/13
52	LF - Annual Monitoring Report - Year 3-2013	385 edays	Wed 5/22/13	Wed 6/11/14
53	LF - Annual Monitoring Report - Year 4-2014	325 edays	Tue 5/20/14	Fri 4/10/15
54	LF - Annual Monitoring Report - Year 5-2015	160 edays	Mon 5/25/15	Sun 11/1/15
55	**LF - Optimized Exit Strategy**	**86 days**	**Wed 5/20/15**	**Thu 9/17/15**
56	LF - Optimized Exit Strategy Document	90 edays	Wed 5/20/15	Tue 8/18/15
57	LF - Acceptance of Optimized Exit Strategy Document	0 days	Thu 9/17/15	Thu 9/17/15
58	**SD031 - Three Mile Creek (Closure Unrestricted Use) Closure Approval 9/2/14.**	**952 days**	**Mon 1/10/11**	**Tue 9/2/14**



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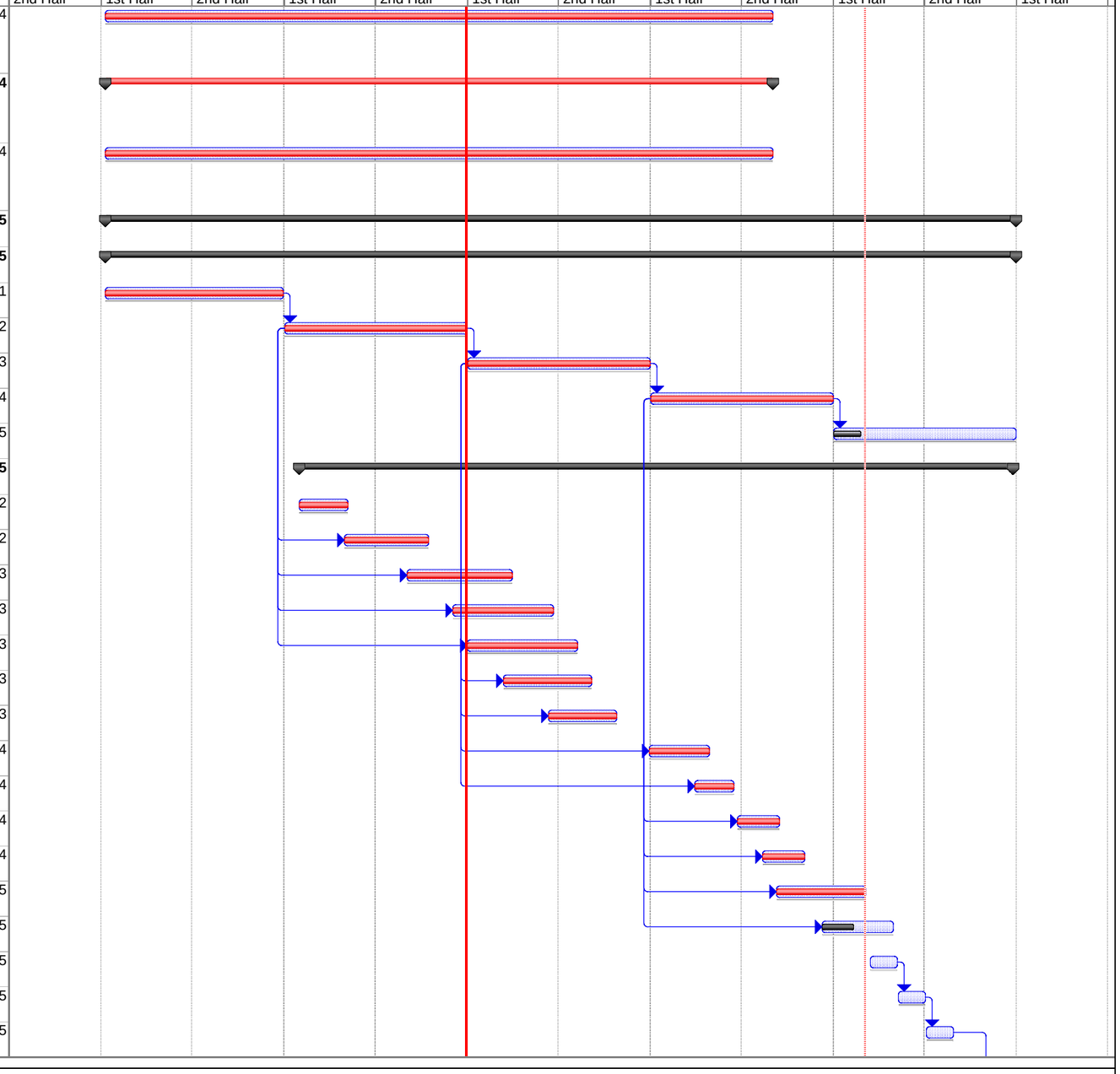
Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York
FA8903-10-D-8595 Delivery Order 0014
Updated 3/6/15

ID	Task Name	Duration	Start	Finish
59	**SD031 - Achieve Site Closure**	952 days	Mon 1/10/11	Tue 9/2/14
60	**SD032 - Six Mile Creek (Closure Unrestricted Use) Closure Approval 9/2/14.**	**952 days**	**Mon 1/10/11**	**Tue 9/2/14**
61	**SD032 - Achieve Site Closure**	952 days	Mon 1/10/11	Tue 9/2/14
62	**SD052 - SVI Systems (Optimized Exit Strategy)**	**1299 days**	**Mon 1/10/11**	**Thu 12/31/15**
63	**SD052 - Field Work**	**1299 days**	**Mon 1/10/11**	**Thu 12/31/15**
64	SD052 - O&M and Monitoring Year 1-2011	355 edays	Mon 1/10/11	Sat 12/31/11
65	SD052 - O&M and Monitoring Year 2-2012	364 edays	Mon 1/2/12	Mon 12/31/12
66	SD052 - O&M and Monitoring Year 3-2013	364 edays	Tue 1/1/13	Tue 12/31/13
67	SD052 - O&M and Monitoring Year 4-2014	364 edays	Wed 1/1/14	Wed 12/31/14
68	SD052 - O&M and Monitoring Year 5-2015	364 edays	Thu 1/1/15	Thu 12/31/15
69	**SD052 - Reporting**	**1018 days**	**Wed 2/1/12**	**Fri 12/25/15**
70	SD052 - O&M and Quarterly Monitoring Report - Year 1 Q4	69 days	Wed 2/1/12	Mon 5/7/12
71	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q1	168 edays	Tue 5/1/12	Tue 10/16/12
72	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q2	210 edays	Mon 9/3/12	Mon 4/1/13
73	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q3	201 edays	Mon 12/3/12	Sat 6/22/13
74	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q4	220 edays	Tue 1/1/13	Fri 8/9/13
75	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q1	176 edays	Wed 3/13/13	Thu 9/5/13
76	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q2	98 days	Wed 6/12/13	Fri 10/25/13
77	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q3	86 days	Mon 12/30/13	Mon 4/28/14
78	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q4	56 days	Mon 3/31/14	Mon 6/16/14
79	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q1	60 days	Tue 6/24/14	Mon 9/15/14
80	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q2	60 days	Wed 8/13/14	Tue 11/4/14
81	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q3	128 days	Wed 9/10/14	Fri 3/6/15
82	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q4	102 days	Wed 12/10/14	Thu 4/30/15
83	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q1	40 days	Mon 3/16/15	Fri 5/8/15
84	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q2	40 days	Mon 5/11/15	Fri 7/3/15
85	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q3	40 days	Mon 7/6/15	Fri 8/28/15



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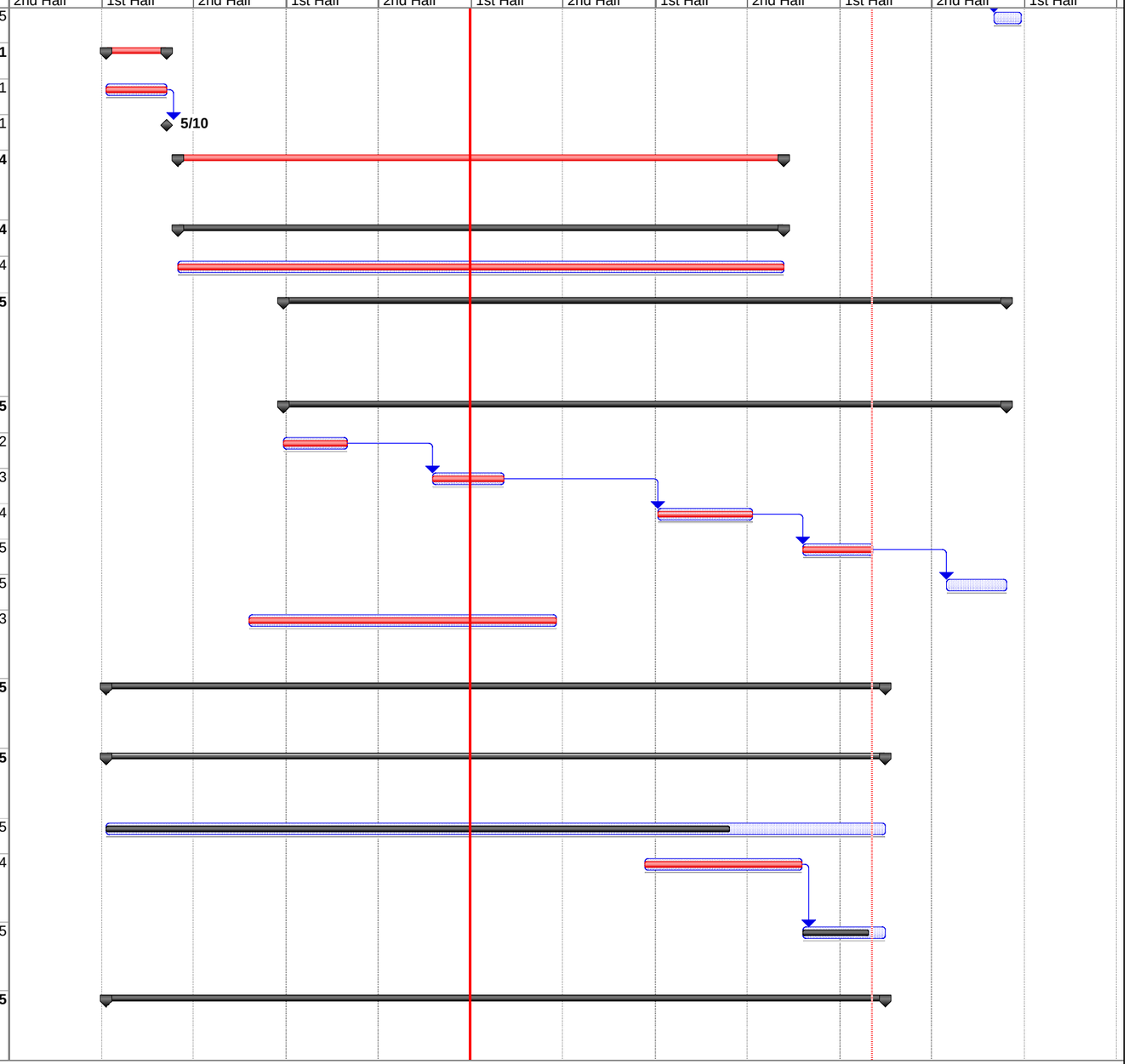
ID	Task Name	Duration	Start	Finish
86	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q4	40 days	Mon 11/2/15	Fri 12/25/15
87	**SD052 - Optimized Exit Strategy**	**86 days**	**Mon 1/10/11**	**Tue 5/10/11**
88	SD052 - Optimized Exit Strategy Document	120 edays	Mon 1/10/11	Tue 5/10/11
89	SD052 - Acceptance of Optimized Exit Strategy Document	0 days	Tue 5/10/11	Tue 5/10/11
90	**SS060 - STW-1300 (Bldg. 35/36) (Closure with Restrictions) Closure Approval 9/11/14)**	**857 days**	**Wed 6/1/11**	**Thu 9/11/14**
91	**SS060 - Site Closure**	**857 days**	**Wed 6/1/11**	**Thu 9/11/14**
92	**SS060 - Site Closure**	857 days	Wed 6/1/11	Thu 9/11/14
93	**Status Quo LUC-IC Sites (ST006, SS024, FT030, SS033, ST036, SS044, SD052-1, -2, -4 &-5, SS062, AOI72, and Building 211)**	**1023 days**	**Tue 12/27/11**	**Fri 11/27/15**
94	**Status Quo CERCLA LUCIC Inspection Sites**	**1023 days**	**Tue 12/27/11**	**Fri 11/27/15**
95	Status Quo Sites - LUCIC Inspection Report - Year 1-2011	90 days	Tue 12/27/11	Mon 4/30/12
96	Status Quo Sites- LUCIC Inspection Report - Year 2-2012	101 days	Wed 10/17/12	Wed 3/6/13
97	Status Quo Sites - LUCIC Inspection Report - Year 3-2013	135 days	Mon 1/6/14	Fri 7/11/14
98	Status Quo Sites - LUCIC Inspection Report - Year 4-2014	100 days	Mon 10/20/14	Fri 3/6/15
99	Status Quo Sites - LUCIC Inspection Report - Year 5-2015	119 edays	Fri 7/31/15	Fri 11/27/15
100	**DP011 - Building 3 (Closure with no Restrictions) Site Closure Approved 6/18/13**	434 days	Thu 10/20/11	Tue 6/18/13
101	**DP012 - Building 301 (Mod 2-Closure with No Restrictions) Closure Pending**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**
102	**DP012 - Unrestricted Site Closure and LUC/IC Inspections until Closure Achieved**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**
103	LUC/IC Inspections and Reporting 2011-2015	1102 days	Mon 1/10/11	Tue 3/31/15
104	DP012 Prepare and Submit Closure Report	223 days	Wed 12/11/13	Fri 10/17/14
105	DP012 - AFCEC & NYSDEC Review and Acceptance of Closure Report	117 days	Mon 10/20/14	Tue 3/31/15
106	**DP013 - Building 255 (Mod 2-Closure with No Restrictions) Closure Pending**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**



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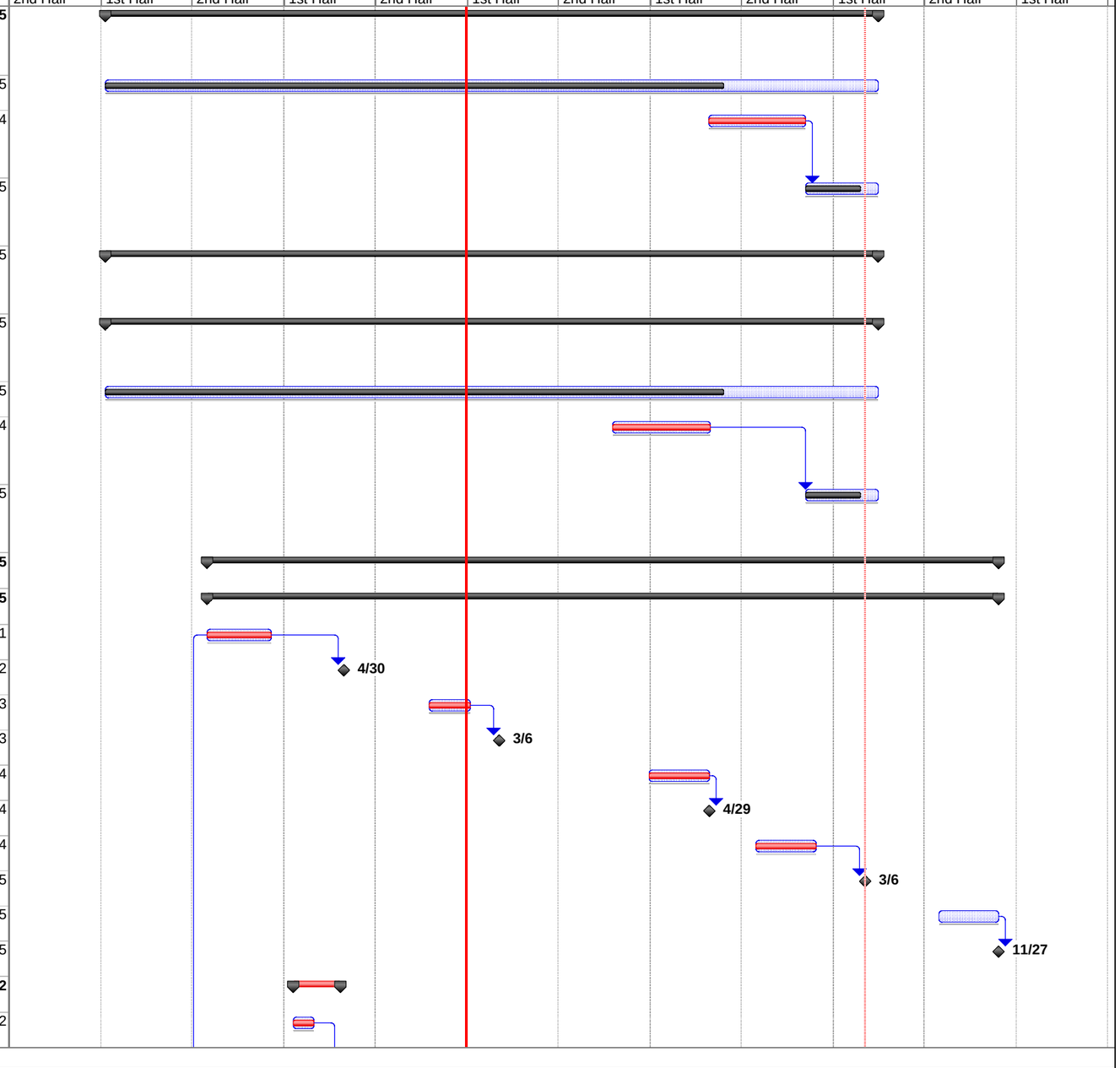
Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York
FA8903-10-D-8595 Delivery Order 0014
Updated 3/6/15

ID	Task Name	Duration	Start	Finish
107	**DP013 - Unrestricted Site Closure and LUC/IC Inspections until Closure Achieved**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**
108	LUC/IC Inspections and Reporting 2011-2015	1102 days	Mon 1/10/11	Tue 3/31/15
109	DP013 Prepare and Submit Closure Report-Addendum Report Submitted Per NYSDEC Request	139 days	Mon 4/28/14	Thu 11/6/14
110	DP013 - AFCEC & NYSDEC Review and Acceptance of Closure Report	103 days	Fri 11/7/14	Tue 3/31/15
111	**DP015 - Building 219 (Mod 2-Closure with No Restrictions) Closure Pending**	1102 days	Mon 1/10/11	Tue 3/31/15
112	**DP015 - Unrestricted Site Closure and LUC/IC Inspections until Closure Achieved**	1102 days	Mon 1/10/11	Tue 3/31/15
113	LUC/IC Inspections and Reporting 2011-2015	1102 days	Mon 1/10/11	Tue 3/31/15
114	DP015 Prepare and Submit Closure Report-Addendum Report Submitted Per NYSDEC Request	139 days	Fri 10/18/13	Wed 4/30/14
115	DP015 - AFCEC & NYSDEC Review and Acceptance of Closure Report	103 days	Fri 11/7/14	Tue 3/31/15
116	**SS017 - Lot 69**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**
117	**SS017 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**
118	SS017 - LUCIC Inspection Report - Year 1-2011	128 edays	Mon 8/1/11	Wed 12/7/11
119	SS017 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Mon 4/30/12	Mon 4/30/12
120	SS017 - LUCIC Inspection Report - Year 2-2012	82 edays	Wed 10/17/12	Mon 1/7/13
121	SS017 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 3/6/13	Wed 3/6/13
122	SS017 - LUCIC Inspection Report - Year 3-2013	120 edays	Mon 12/30/13	Tue 4/29/14
123	SS017 - Acceptance of LUCIC Inspection Report	0 days	Tue 4/29/14	Tue 4/29/14
124	SS017 - LUCIC Inspection Report - Year 4-2014	120 edays	Thu 7/31/14	Fri 11/28/14
125	SS017 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 3/6/15	Fri 3/6/15
126	SS017 - LUCIC Inspection Report - Year 5-2015	119 edays	Fri 7/31/15	Fri 11/27/15
127	SS017 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Fri 11/27/15	Fri 11/27/15
128	**SS017 - Removal of Groundwater LUCIC**	**66 days**	**Fri 1/20/12**	**Mon 4/23/12**
129	SS017 - Groundwater LUCIC Removal Documentation Report	41 edays	Fri 1/20/12	Thu 3/1/12



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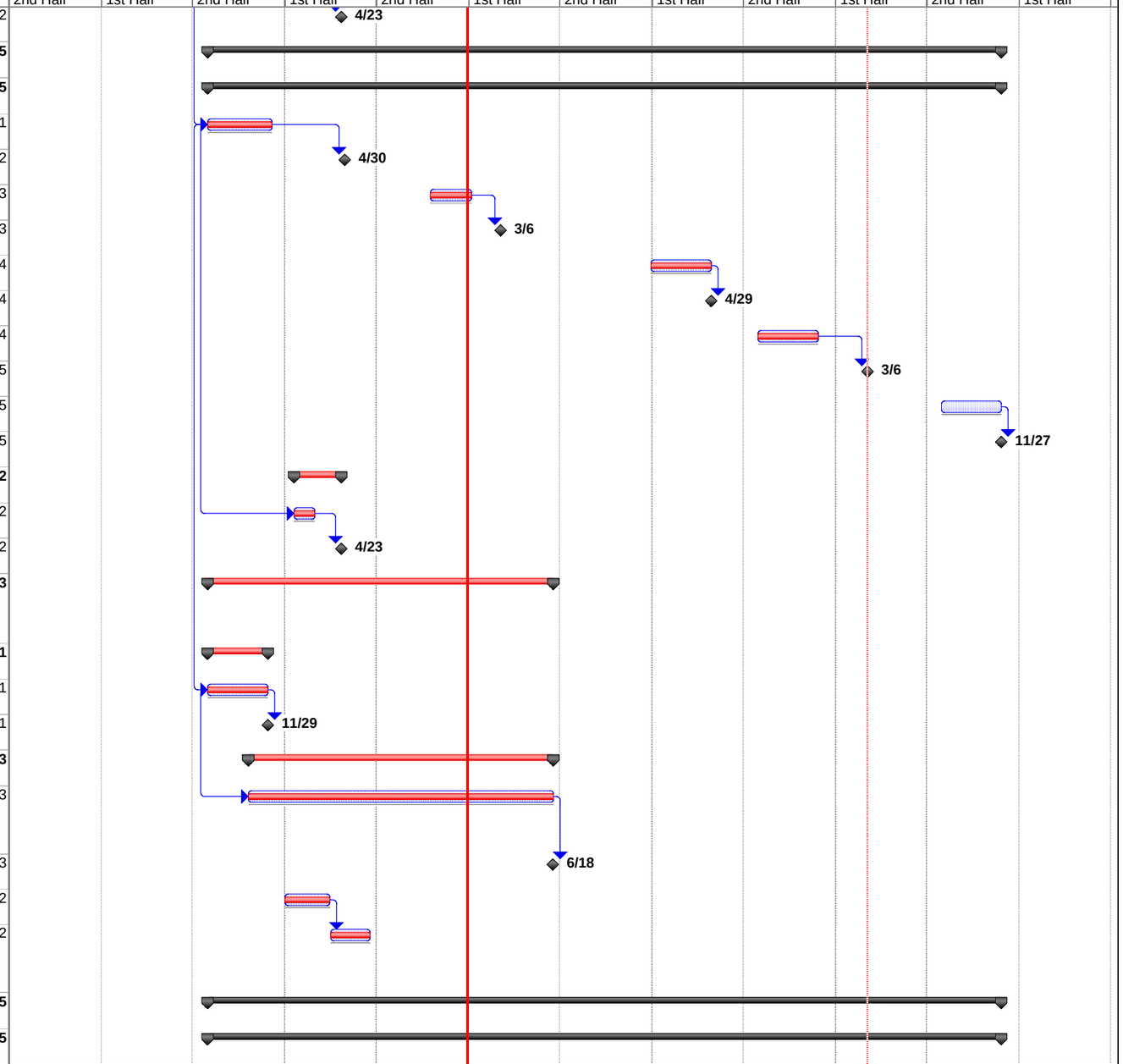
ID	Task Name	Duration	Start	Finish
130	SS017 - Acceptance of LUCIC Removal	0 days	Mon 4/23/12	Mon 4/23/12
131	**DP022 - Building 222**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**
132	**DP022 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**
133	DP022 - LUCIC Inspection Report - Year 1-2011	128 edays	Mon 8/1/11	Wed 12/7/11
134	DP022 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Mon 4/30/12	Mon 4/30/12
135	DP022 - LUCIC Inspection Report - Year 2-2012	82 edays	Wed 10/17/12	Mon 1/7/13
136	DP022 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 3/6/13	Wed 3/6/13
137	DP022 - LUCIC Inspection Report - Year 3-2013	120 edays	Mon 12/30/13	Tue 4/29/14
138	DP022 - Acceptance of LUCIC Inspection Report - Year 3	0 days	Tue 4/29/14	Tue 4/29/14
139	DP022 - LUCIC Inspection Report - Year 4-2014	120 edays	Thu 7/31/14	Fri 11/28/14
140	DP022 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 3/6/15	Fri 3/6/15
141	DP022 - LUCIC Inspection Report - Year 5-2015	119 edays	Fri 7/31/15	Fri 11/27/15
142	DP022 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Fri 11/27/15	Fri 11/27/15
143	**DP022 - Removal of Groundwater LUCIC**	**66 days**	**Fri 1/20/12**	**Mon 4/23/12**
144	DP022 - Groundwater LUCIC Removal Documentation Report	41 edays	Fri 1/20/12	Thu 3/1/12
145	DP022 - Acceptance of LUCIC Removal	0 days	Mon 4/23/12	Mon 4/23/12
146	**SS023 - Building 20 (Closure with no Restrictions) Closure Approval 6/18/13**	**492 days**	**Mon 8/1/11**	**Tue 6/18/13**
147	**SS023 - LUCIC Inspection**	**86 days**	**Mon 8/1/11**	**Tue 11/29/11**
148	SS023 - LUCIC Inspection Report - Year 1	120 edays	Mon 8/1/11	Tue 11/29/11
149	SS023 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Tue 11/29/11	Tue 11/29/11
150	**SS023 - Removal of LUCIC**	**433 days**	**Thu 10/20/11**	**Tue 6/18/13**
151	SS023 - Soil and Groundwater Sampling/Reporting/Closure Report	607 edays	Thu 10/20/11	Tue 6/18/13
152	SS023 - Acceptance of LUCIC Removal	0 days	Tue 6/18/13	Tue 6/18/13
153	SS023 Prepare and Submit Closure Report	65 days	Mon 1/2/12	Fri 3/30/12
154	SS023 - AFCEC & NYSDEC Review and Acceptance of Closure Report	56 days	Mon 4/2/12	Mon 6/18/12
155	**SS025 - Site T-9**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**
156	**SS025 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Fri 11/27/15**



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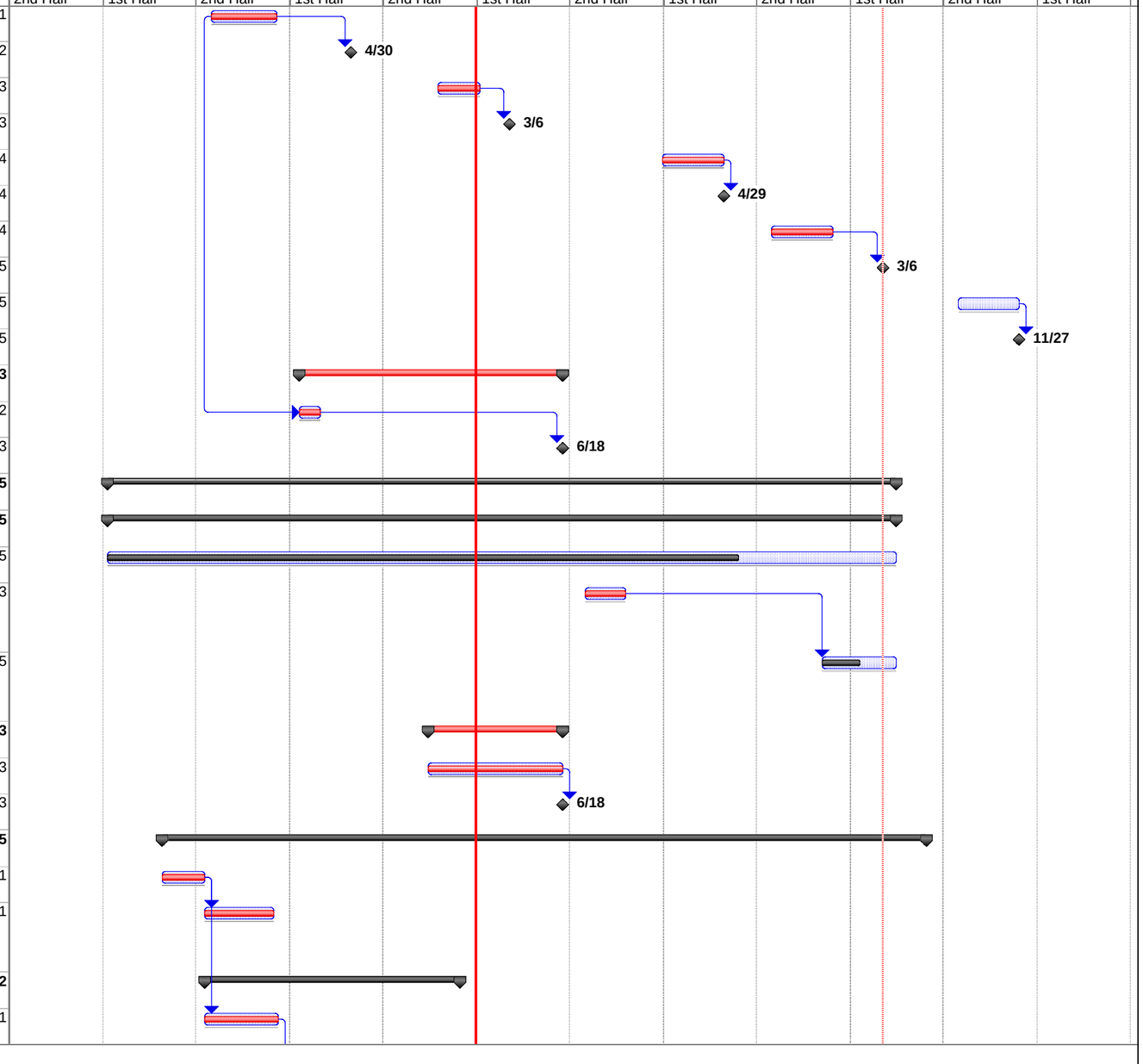
157	SS025 - LUCIC Inspection Report - Year 1	128 edays	Mon 8/1/11	Wed 12/7/11
158	SS025 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Mon 4/30/12	Mon 4/30/12
159	SS025 - LUCIC Inspection Report - Year 2	82 edays	Wed 10/17/12	Mon 1/7/13
160	SS025 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 3/6/13	Wed 3/6/13
161	SS025 - LUCIC Inspection Report - Year 3	120 edays	Mon 12/30/13	Tue 4/29/14
162	SS025 - Acceptance of LUCIC Inspection Report - Year 3	0 days	Tue 4/29/14	Tue 4/29/14
163	SS025 - LUCIC Inspection Report - Year 4	120 edays	Thu 7/31/14	Fri 11/28/14
164	SS025 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 3/6/15	Fri 3/6/15
165	SS025 - LUCIC Inspection Report - Year 5	119 edays	Fri 7/31/15	Fri 11/27/15
166	SS025 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Fri 11/27/15	Fri 11/27/15
167	**SS025 - Removal of Groundwater LUCIC**	**367 days**	**Fri 1/20/12**	**Tue 6/18/13**
168	SS025 - Groundwater LUCIC Removal Documentation Report	41 edays	Fri 1/20/12	Thu 3/1/12
169	SS025 - Acceptance of LUCIC Removal	0 days	Tue 6/18/13	Tue 6/18/13
170	**SD050 - Building 214 (Mod 2-Closure with No Restrictions)**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**
171	**SD050 - LUCIC Inspection**	**1102 days**	**Mon 1/10/11**	**Tue 3/31/15**
172	LUC/IC Inspections and Reporting 2011-2015	1102 days	Mon 1/10/11	Tue 3/31/15
173	DP015 Prepare and Submit Closure Report-Addendum Report Submitted Per NYSDEC Request	57 days	Thu 8/1/13	Fri 10/18/13
174	DP015 - AFCEC & NYSDEC Review and Acceptance of Closure Report	103 days	Fri 11/7/14	Tue 3/31/15
175	**SD050 - Removal of Groundwater LUCIC**	**187 days**	**Fri 9/28/12**	**Tue 6/18/13**
176	SD050 - Groundwater LUCIC Removal Documentation Report	263 edays	Fri 9/28/12	Tue 6/18/13
177	SD050 - Acceptance of LUCIC Removal	0 days	Tue 6/18/13	Tue 6/18/13
178	**SD041 - Nose Doc #1 (Closure Unrestricted Reuse)**	**1068 days**	**Wed 4/27/11**	**Fri 5/29/15**
179	SD041 -Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
180	SD041 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
181	**SD041 - Conduct Monitoring, Operations & Maintenance**	**357 days**	**Tue 7/19/11**	**Wed 11/28/12**
182	SD041 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM	104 days	Tue 7/19/11	Fri 12/9/11



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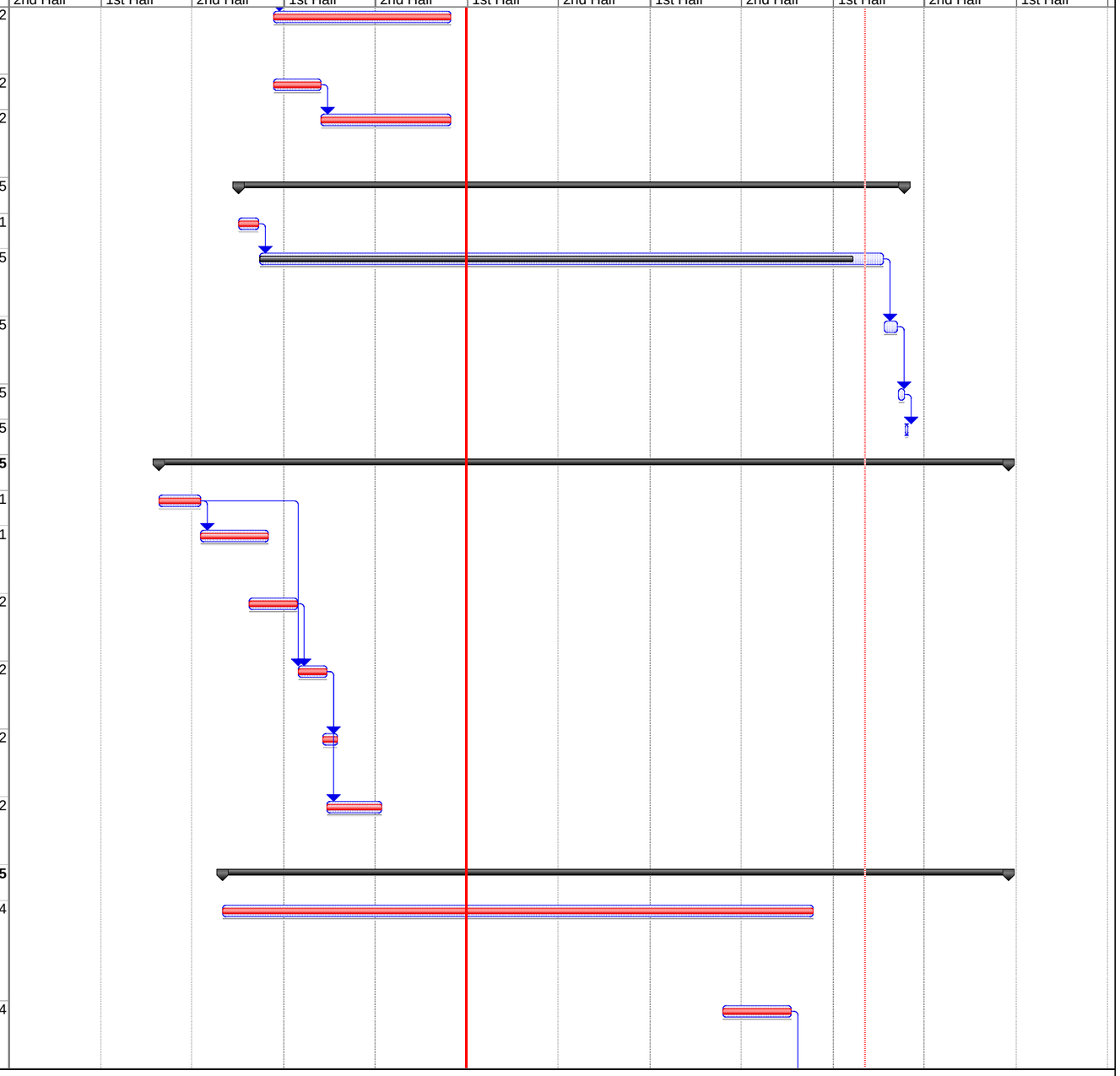
ID	Task Name	Duration	Start	Finish
183	SD041 - AFCEC & NYSDEC Review and Acceptance of CY11 Q2 LTO/LTM Report	253 days	Mon 12/12/11	Wed 11/28/12
184	SD041 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM	68 days	Mon 12/12/11	Wed 3/14/12
185	SD041 - AFCEC & NYSDEC Review and Acceptance of CY11 Q3 LTO/LTM Report	185 days	Thu 3/15/12	Wed 11/28/12
186	**SD041 - Achieve Site Closure**	950 days	Mon 10/3/11	Fri 5/22/15
187	SD041 - Prepare and Submit Closure Report	30 days	Mon 10/3/11	Fri 11/11/11
188	SD041 - Prepare and Submit Closure Report Addendum Report required to address additional sampling request.	890 days	Mon 11/14/11	Fri 4/10/15
189	SD041 - AFCEC & NYSDEC Review and Acceptance of Closure Report	20 days	Mon 4/13/15	Fri 5/8/15
190	SD041 - NYSDEC Closure of Spill Number	10 days	Mon 5/11/15	Fri 5/22/15
191	SD041 - Wells Decommissioned	5 days	Mon 5/25/15	Fri 5/29/15
192	**SS054 - BLDG 781 Pumphouse (Optimized Exit Strategy)**	**1211 days**	**Wed 4/27/11**	**Wed 12/16/15**
193	SS054 - Design/Plan Remedy Optimization	59 days	Wed 4/27/11	Mon 7/18/11
194	SS054 - AFCEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
195	SS054 - Complete Supplemental Investigation, submit Investigation Report	70 days	Mon 10/24/11	Fri 1/27/12
196	SS054 - Implement Remedy Optimization Systems and Submit Report	41 days	Mon 1/30/12	Mon 3/26/12
197	SS054 - AFCEC & NYSDEC Review and Acceptance of Investigation Report	21 days	Mon 3/19/12	Mon 4/16/12
198	SS054 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	79 days	Tue 3/27/12	Fri 7/13/12
199	**SS054 - Conduct Monitoring, Operations & Maintenance**	**1120 days**	**Thu 9/1/11**	**Wed 12/16/15**
200	SS054 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports for 2011, CY2012, 2013 and Receive Regulatory Acceptance	842 days	Thu 9/1/11	Fri 11/21/14
201	SS054 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	98 days	Mon 5/26/14	Wed 10/8/14



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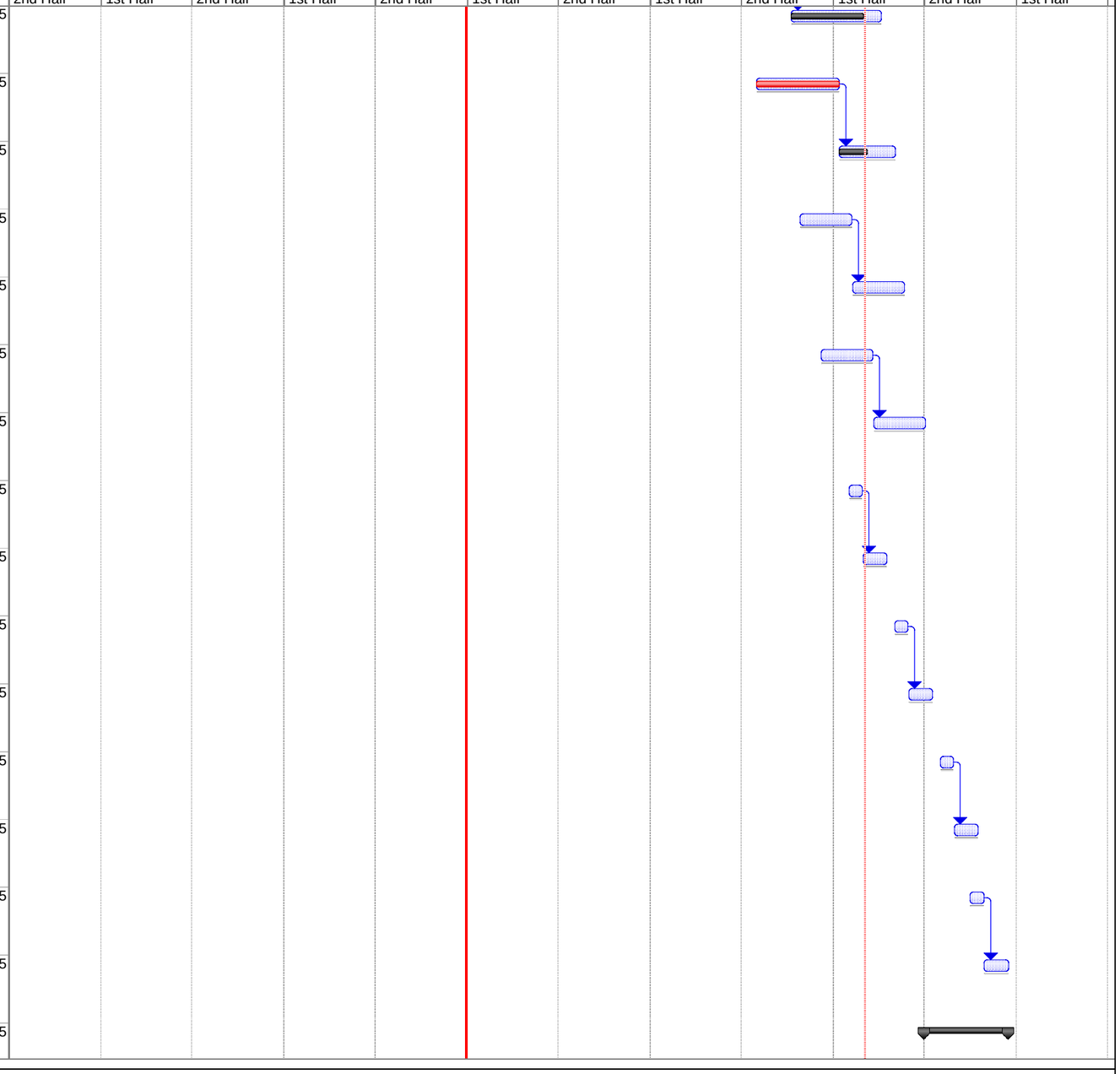
202	SS054 - AFCEC & NYSDEC Review and Acceptance of CY14 Q1 LTO/LTM Report	128 days	Thu 10/9/14	Mon 4/6/15
203	SS054 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	117 days	Fri 8/1/14	Mon 1/12/15
204	SS054 - AFCEC & NYSDEC Review and Acceptance of CY14 Q2 LTO/LTM Report	80 days	Tue 1/13/15	Mon 5/4/15
205	SS054 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	75 days	Mon 10/27/14	Fri 2/6/15
206	SS054 - AFCEC & NYSDEC Review and Acceptance of CY14 Q3 LTO/LTM Report	75 days	Mon 2/9/15	Fri 5/22/15
207	SS054 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	75 days	Mon 12/8/14	Fri 3/20/15
208	SS054 - AFCEC & NYSDEC Review and Acceptance of CY14 Q4 LTO/LTM Report	75 days	Mon 3/23/15	Fri 7/3/15
209	SS054 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	20 days	Mon 2/2/15	Fri 2/27/15
210	SS054 - AFCEC & NYSDEC Review and Acceptance of CY15 Q1 LTO/LTM Report	35 days	Mon 3/2/15	Fri 4/17/15
211	SS054 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	20 days	Mon 5/4/15	Fri 5/29/15
212	SS054 - AFCEC & NYSDEC Review and Acceptance of CY15 Q2 LTO/LTM Report	35 days	Mon 6/1/15	Fri 7/17/15
213	SS054 - Conduct Monitoring, Prep and Submit CY15 Q3 LTO/LTM Report	20 days	Mon 8/3/15	Fri 8/28/15
214	SS054 - AFCEC & NYSDEC Review and Acceptance of CY15 Q3 LTO/LTM Report	35 days	Mon 8/31/15	Fri 10/16/15
215	SS054 - Conduct Monitoring, Prep and Submit CY15 Q4 LTO/LTM Report	20 days	Thu 10/1/15	Wed 10/28/15
216	SS054 - AFCEC & NYSDEC Review and Acceptance of CY15 Q4 LTO/LTM Report	35 days	Thu 10/29/15	Wed 12/16/15
217	**SS054 - Optimization Exit Strategy**	120 days	Wed 7/1/15	Tue 12/15/15



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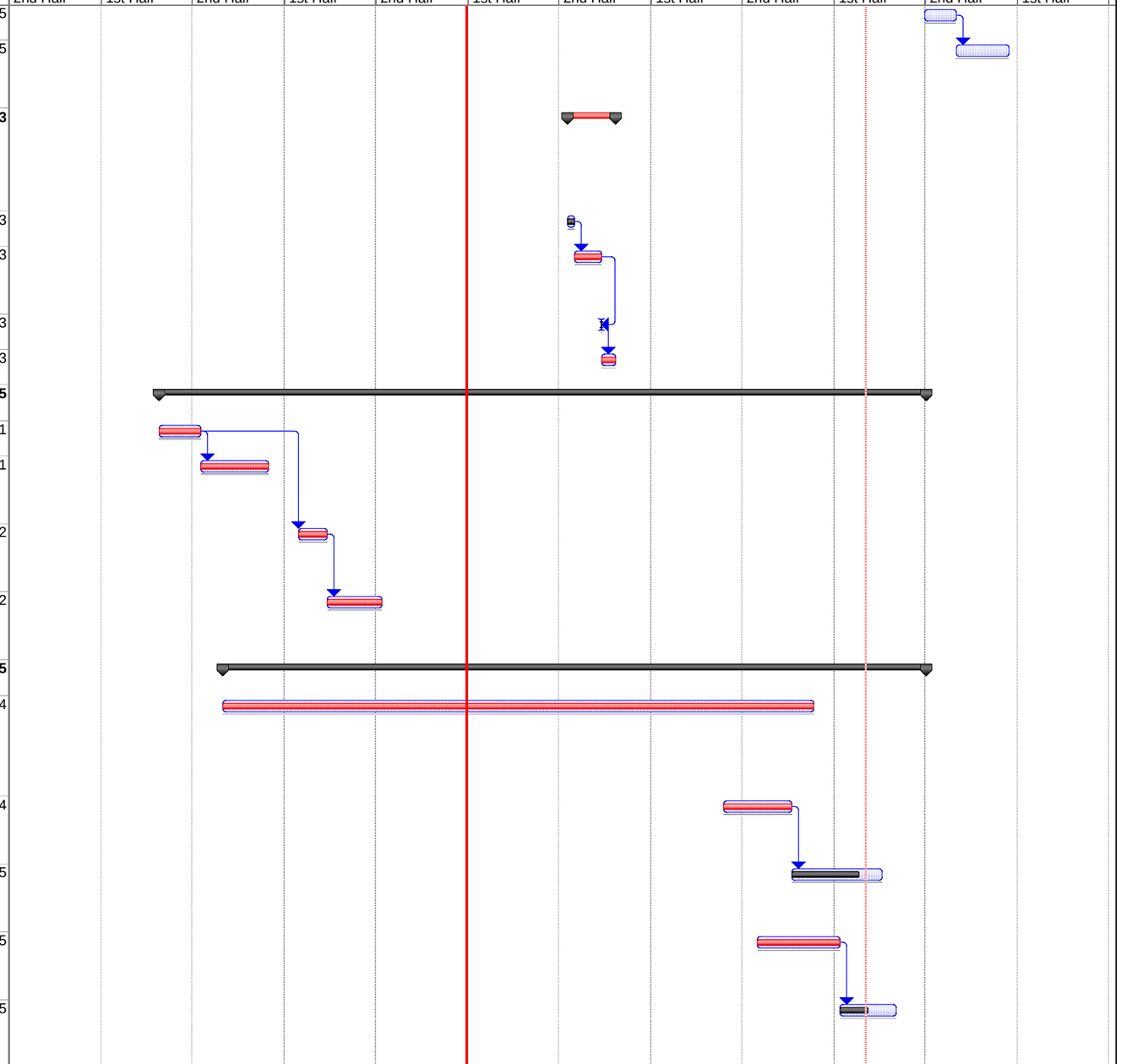
ID	Task Name	Duration	Start	Finish
218	SS054 - Prepare and Submit Optimized Exit Strategy Report	45 days	Wed 7/1/15	Tue 9/1/15
219	SS054 - AFCEC & NYSDEC Review and Acceptance of Optimized Exit Strategy Report	75 days	Wed 9/2/15	Tue 12/15/15
220	**SS020 - Tank Farms 1 & 3 (Closure Unrestricted Reuse) Revised approach to LTM Closure by 2015. Closure Approval 9/25/13.**	**68 days**	**Fri 7/19/13**	**Tue 10/22/13**
221	SS020 - Prepare and Submit Closure Report	10 days	Fri 7/19/13	Thu 8/1/13
222	SS020 - AFCEC & NYSDEC Review and Acceptance of Closure Report	38 days	Fri 8/2/13	Tue 9/24/13
223	SS020 - NYSDEC Closure of Spill Number	1 day	Tue 9/24/13	Tue 9/24/13
224	SS020 - Wells Decommissioned	20 days	Wed 9/25/13	Tue 10/22/13
225	**SS063 - Apron 1 (Closure Unrestricted Reuse)**	**1093 days**	**Wed 4/27/11**	**Fri 7/3/15**
226	SS063 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
227	SS063 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
228	SS063 - Implement Remedy Optimization Systems and Submit Report	41 days	Mon 1/30/12	Mon 3/26/12
229	SS063 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	79 days	Tue 3/27/12	Fri 7/13/12
230	**SS063 - Conduct Monitoring, Operations & Maintenance**	**1002 days**	**Thu 9/1/11**	**Fri 7/3/15**
231	SS063 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports for 2011, 2012, 2013 and Achieve AFCEC and Regulatory Acceptance	842 days	Thu 9/1/11	Fri 11/21/14
232	SS063 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	98 days	Mon 5/26/14	Wed 10/8/14
233	SS063 - AFCEC & NYSDEC Review and Acceptance of CY14 Q1 LTO/LTM Report	128 days	Thu 10/9/14	Mon 4/6/15
234	SS063 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	117 days	Fri 8/1/14	Mon 1/12/15
235	SS063 - AFCEC & NYSDEC Review and Acceptance of CY14 Q2 LTO/LTM Report	80 days	Tue 1/13/15	Mon 5/4/15



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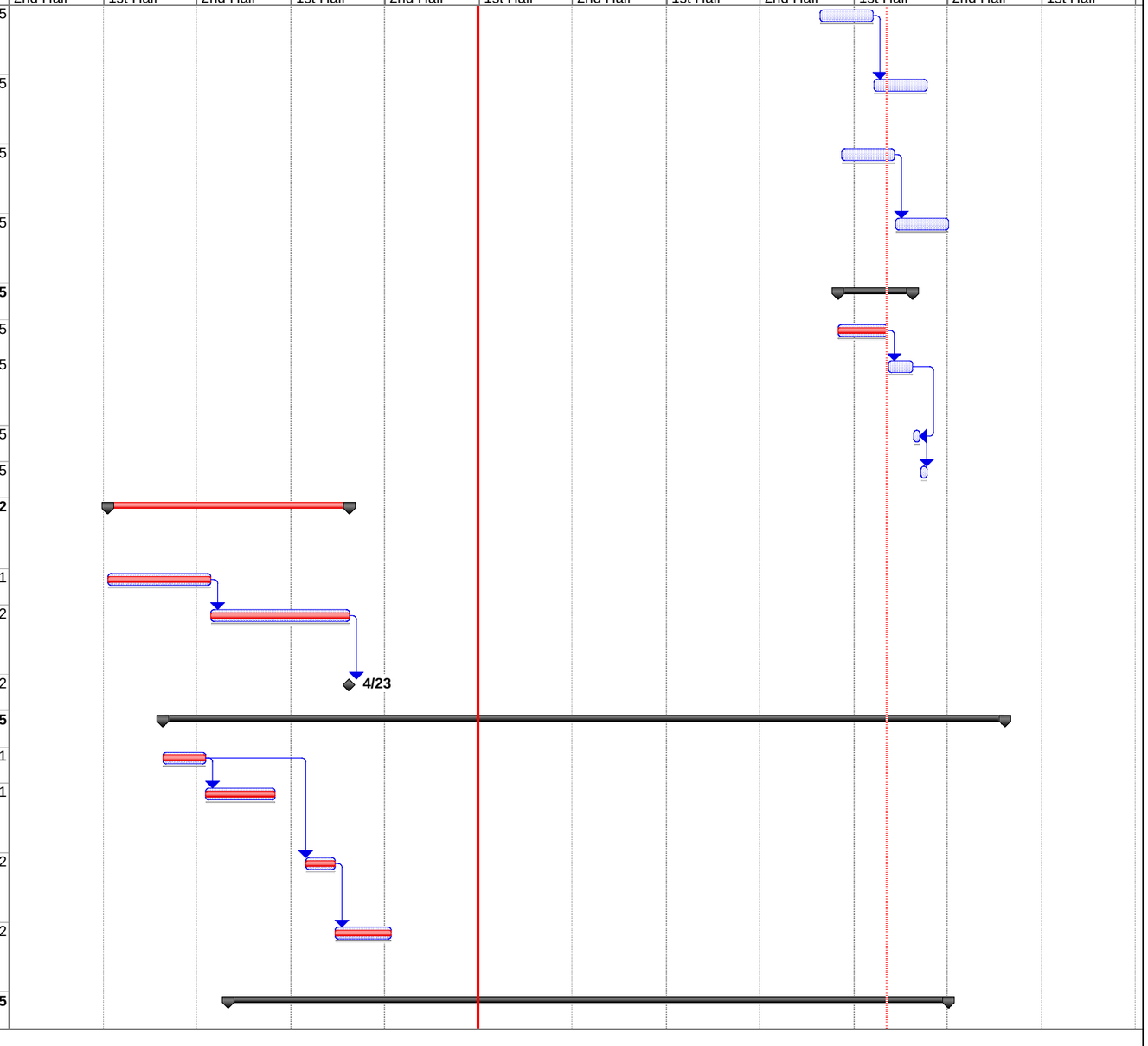
ID	Task Name	Duration	Start	Finish
236	SS063 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	75 days	Mon 10/27/14	Fri 2/6/15
237	SS063 - AFCEC & NYSDEC Review and Acceptance of CY14 Q3 LTO/LTM Report	75 days	Mon 2/9/15	Fri 5/22/15
238	SS063 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	75 days	Mon 12/8/14	Fri 3/20/15
239	SS063 - AFCEC & NYSDEC Review and Acceptance of CY14 Q4 LTO/LTM Report	75 days	Mon 3/23/15	Fri 7/3/15
240	**SS063 - Prepare and Submit Closure Report**	**105 days**	**Mon 12/1/14**	**Fri 4/24/15**
241	Prepare and Submit Draft Closure Report	70 days	Mon 12/1/14	Fri 3/6/15
242	SS063 - AFCEC & NYSDEC Review and Acceptance of Closure Report	35 days	Mon 3/9/15	Fri 4/24/15
243	SS063 - NYSDEC Closure of Spill Number	10 days	Mon 4/27/15	Fri 5/8/15
244	SS063 - Wells Decommissioned	10 days	Mon 5/11/15	Fri 5/22/15
245	**ST037 - Bldg 771, Pumphouse 5 (Closure Unrestricted Use) Closure Approval 4/23/12**	**336 days**	**Mon 1/10/11**	**Mon 4/23/12**
246	ST037 - Prepare and Submit Closure Report	144 days	Mon 1/10/11	Thu 7/28/11
247	ST037 - AFCEC & NYSDEC Review and Acceptance of Closure Report	192 days	Fri 7/29/11	Mon 4/23/12
248	ST037 - NYSDEC Closure of Spill Number	0 days	Mon 4/23/12	Mon 4/23/12
249	**SS064 - Apron 2 (Closure Unrestricted Reuse)**	**1171 days**	**Wed 4/27/11**	**Wed 10/21/15**
250	SS064 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
251	SS064 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
252	SS064 - Implement Remedy Optimization Systems and Submit Report	41 days	Mon 1/30/12	Mon 3/26/12
253	SS064 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	79 days	Tue 3/27/12	Fri 7/13/12
254	**SS064 - Conduct Monitoring, Operations & Maintenance**	**1002 days**	**Thu 9/1/11**	**Fri 7/3/15**



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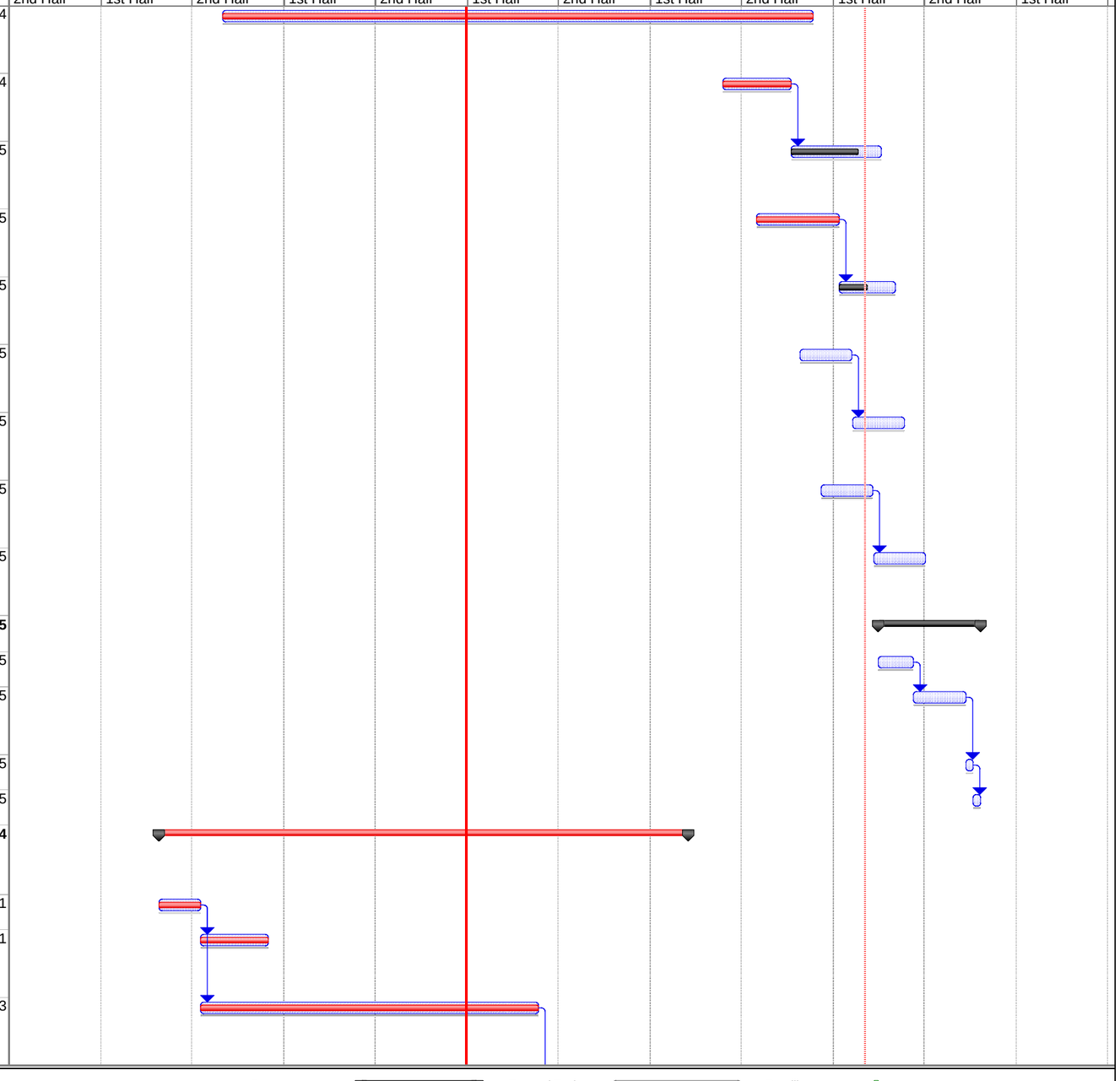
255	SS064 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports for 2011, 2012, 2013 and Achieve AFCEC and Regulatory Acceptance	842 days	Thu 9/1/11	Fri 11/21/14
256	SS064 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	98 days	Mon 5/26/14	Wed 10/8/14
257	SS064 - AFCEC & NYSDEC Review and Acceptance of CY14 Q1 LTO/LTM Report	128 days	Thu 10/9/14	Mon 4/6/15
258	SS064 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	117 days	Fri 8/1/14	Mon 1/12/15
259	SS064 - AFCEC & NYSDEC Review and Acceptance of CY14 Q2 LTO/LTM Report	80 days	Tue 1/13/15	Mon 5/4/15
260	SS064 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	75 days	Mon 10/27/14	Fri 2/6/15
261	SS064 - AFCEC & NYSDEC Review and Acceptance of CY14 Q3 LTO/LTM Report	75 days	Mon 2/9/15	Fri 5/22/15
262	SS064 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	75 days	Mon 12/8/14	Fri 3/20/15
263	SS064 - AFCEC & NYSDEC Review and Acceptance of CY14 Q4 LTO/LTM Report	75 days	Mon 3/23/15	Fri 7/3/15
264	**SS064 - Closure Report**	**146 days**	**Wed 4/1/15**	**Wed 10/21/15**
265	SS064 - Prepare and Submit Closure Report	50 days	Wed 4/1/15	Tue 6/9/15
266	SS064 - AFCEC & NYSDEC Review and Acceptance of Closure Report	75 days	Wed 6/10/15	Tue 9/22/15
267	SS064 - NYSDEC Closure of Spill Number	10 days	Wed 9/23/15	Tue 10/6/15
268	SS064 - Wells Decommissioned	11 days	Wed 10/7/15	Wed 10/21/15
269	**SS065 - BLDG 15 (Closure Unrestricted Reuse) Closure Approval 1/8/14**	**754 days**	**Wed 4/27/11**	**Mon 3/17/14**
270	SS065 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
271	SS065 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
272	SS065 - Implement Remedy Optimization Systems and Submit Report	482 days	Tue 7/19/11	Wed 5/22/13



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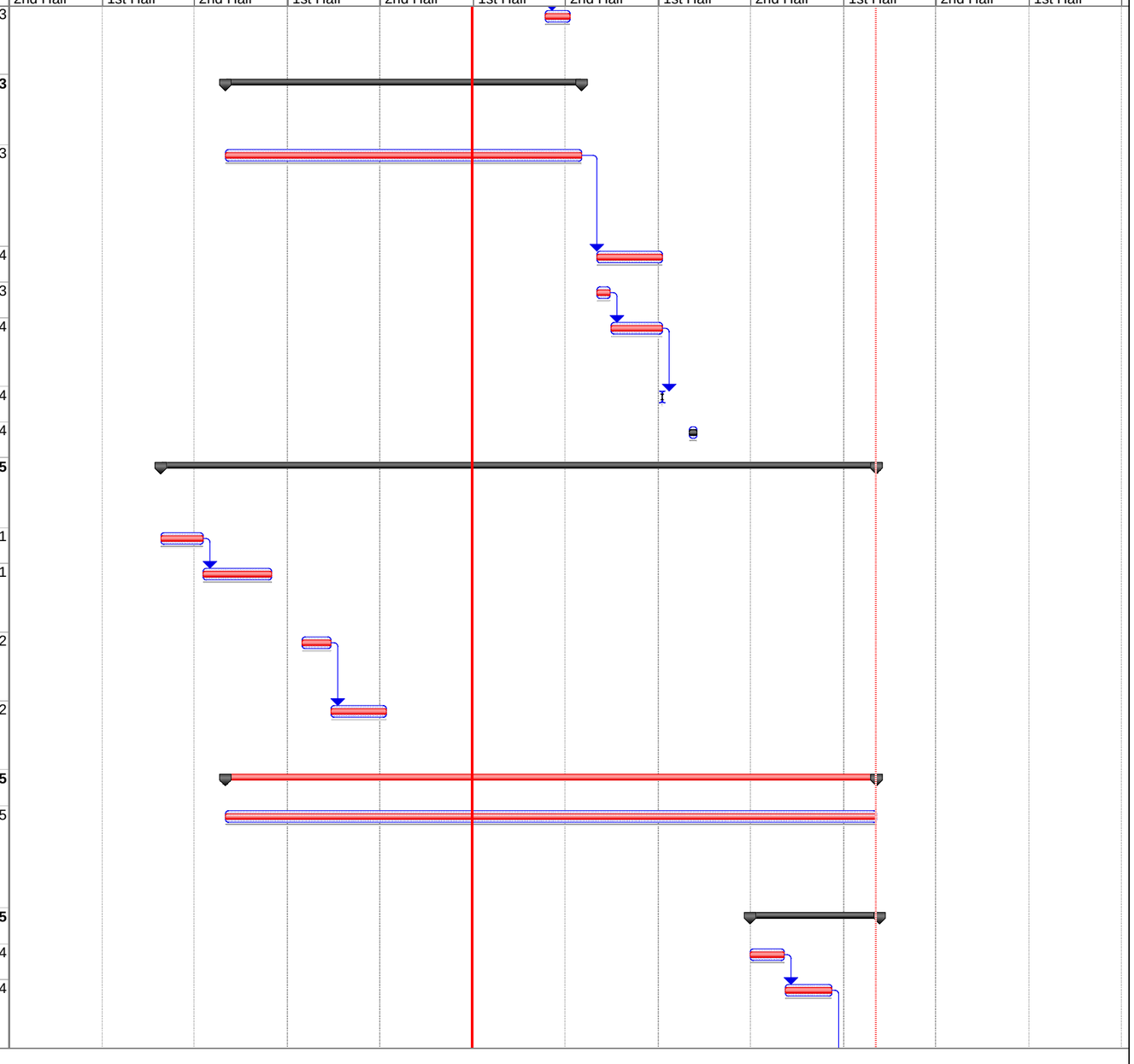
273	SS065 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	35 days	Thu 5/23/13	Wed 7/10/13
274	**SS065 - Conduct Monitoring, Operations & Maintenance**	**502 days**	**Thu 9/1/11**	**Fri 8/2/13**
275	SS065 - Conduct Monitoring, Prep and Submit QuarterlyLTO/LTM Reports 2011, 2012 and Achieve AFCEC and NYSDEC Approval	502 days	Thu 9/1/11	Fri 8/2/13
276	SS065 - Achieve Site Closure	93 days	Mon 9/2/13	Wed 1/8/14
277	SS065 - Prepare and Submit Closure Report	20 days	Mon 9/2/13	Fri 9/27/13
278	SS065 - AFCEC & NYSDEC Review and Acceptance of Closure Report	73 days	Mon 9/30/13	Wed 1/8/14
279	SS065 - NYSDEC Closure of Spill Number	1 day	Wed 1/8/14	Wed 1/8/14
280	SS065 - Wells Decommissioned	11 days	Mon 3/3/14	Mon 3/17/14
281	**SS066 - BLDG 786 (Closure Unrestricted Reuse) Closure Approval 12/8/14**	**1008 days**	**Wed 4/27/11**	**Fri 3/6/15**
282	SS066 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
283	SS066 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
284	SS066 - Implement Remedy Optimization Systems and Submit Report	41 days	Mon 1/30/12	Mon 3/26/12
285	SS066 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	79 days	Tue 3/27/12	Fri 7/13/12
286	**SS066 - Conduct Monitoring, Operations & Maintenance**	**917 days**	**Thu 9/1/11**	**Fri 3/6/15**
287	SS065 - Conduct Monitoring, Prep and Submit QuarterlyLTO/LTM Reports 2011, 2012, 2013,2014 QTR 1 and Achieve AFCEC and NYSDEC Approval	917 days	Thu 9/1/11	Fri 3/6/15
288	**SS066 - Achieve Site Closure**	**183 days**	**Tue 7/1/14**	**Thu 3/12/15**
289	SS066 - Prepare and Submit Closure Report	49 days	Tue 7/1/14	Fri 9/5/14
290	SS066 - AFCEC & NYSDEC Review and Acceptance of Closure Report	66 days	Mon 9/8/14	Mon 12/8/14



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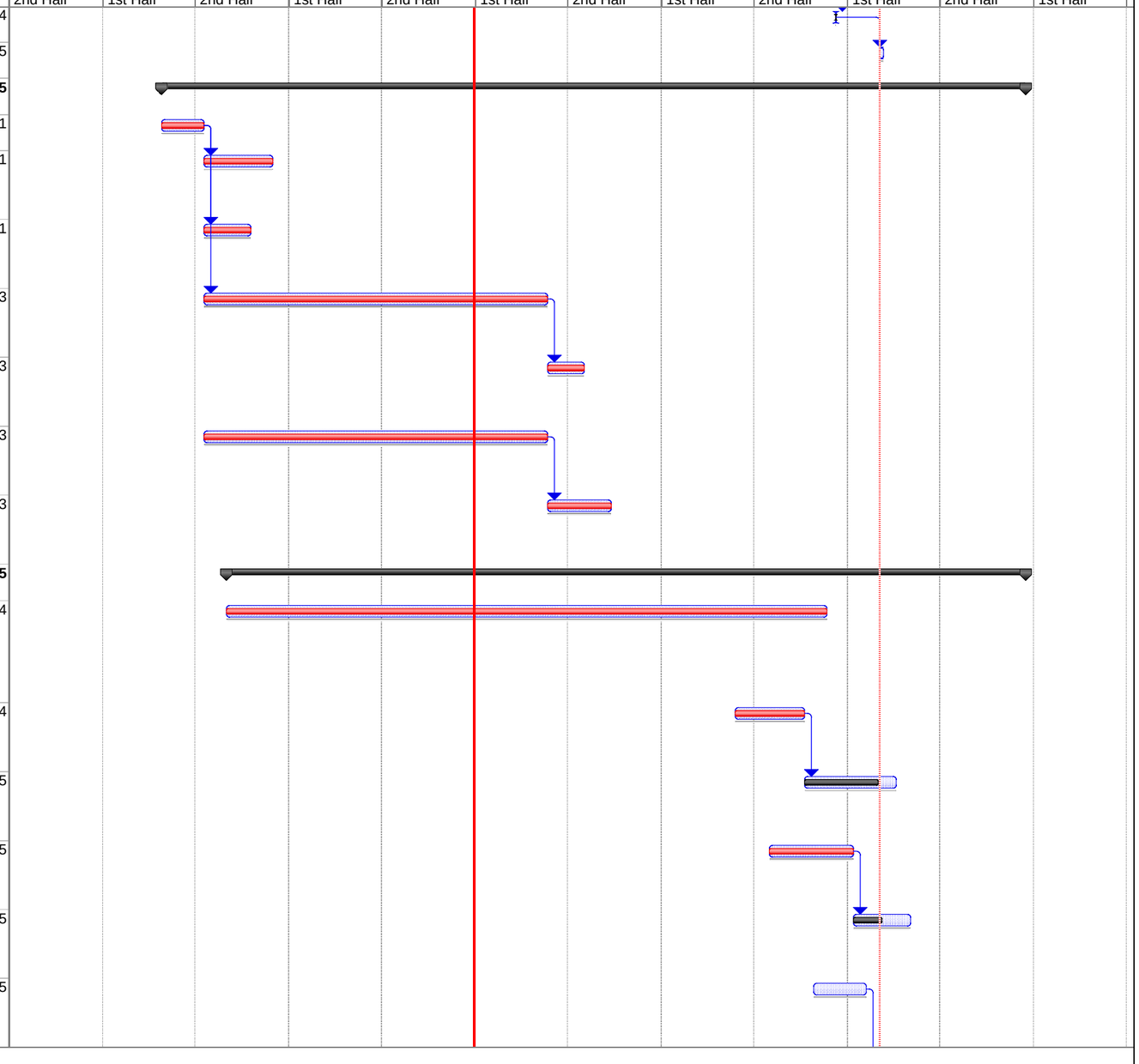
291	SS066 - NYSDEC Closure of Spill Number	1 day	Tue 12/9/14	Tue 12/9/14
292	SS066 - Wells Decommissioned	5 days	Fri 3/6/15	Thu 3/12/15
293	**SS067 - BLDG 789 (Optimized Exit Strategy)**	**1211 days**	**Wed 4/27/11**	**Wed 12/16/15**
294	SS067 - Design/Plan Remedy Optimation	59 days	Wed 4/27/11	Mon 7/18/11
295	SS067 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
296	SS067 - Complete Supplemental Investigation, Submit Investigation Report	66 days	Tue 7/19/11	Tue 10/18/11
297	SS067 - Implement Remedy Optimization Systems and Submit Report	482 days	Tue 7/19/11	Wed 5/22/13
298	SS067 - AFCEC & NYSDEC Review and Acceptance of Investigation Report	52 days	Thu 5/23/13	Fri 8/2/13
299	SS067 - Implement Remedy Optimization Systems and Submit Report	482 days	Tue 7/19/11	Wed 5/22/13
300	SS067 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	89 days	Thu 5/23/13	Tue 9/24/13
301	**SS067 - Conduct Monitoring, Operations & Maintenance**	**1120 days**	**Thu 9/1/11**	**Wed 12/16/15**
302	SS067 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports 2011 Q2, 2012, 2013 and Achieve AFCEC and NYSDEC Approval.	842 days	Thu 9/1/11	Fri 11/21/14
303	SS067 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	98 days	Mon 5/26/14	Wed 10/8/14
304	SS067 - AFCEC & NYSDEC Review and Acceptance of CY14 Q1 LTO/LTM Report	128 days	Thu 10/9/14	Mon 4/6/15
305	SS067 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	117 days	Fri 8/1/14	Mon 1/12/15
306	SS067 - AFCEC & NYSDEC Review and Acceptance of CY14 Q2 LTO/LTM Report	80 days	Tue 1/13/15	Mon 5/4/15
307	SS067 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	75 days	Mon 10/27/14	Fri 2/6/15



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308	SS067 - AFCEC & NYSDEC Review and Acceptance of CY14 Q3 LTO/LTM Report	75 days	Mon 2/9/15	Fri 5/22/15
309	SS067 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	75 days	Mon 12/8/14	Fri 3/20/15
310	SS067 - AFCEC & NYSDEC Review and Acceptance of CY14 Q4 LTO/LTM Report	75 days	Mon 3/23/15	Fri 7/3/15
311	SS067 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	20 days	Mon 2/2/15	Fri 2/27/15
312	SS067 - AFCEC & NYSDEC Review and Acceptance of CY15 Q1 LTO/LTM Report	35 days	Mon 3/2/15	Fri 4/17/15
313	SS067 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	20 days	Mon 5/4/15	Fri 5/29/15
314	SS067 - AFCEC & NYSDEC Review and Acceptance of CY15 Q2 LTO/LTM Report	35 days	Mon 6/1/15	Fri 7/17/15
315	SS067 - Conduct Monitoring, Prep and Submit CY15 Q3 LTO/LTM Report	20 days	Mon 8/3/15	Fri 8/28/15
316	SS067 - AFCEC & NYSDEC Review and Acceptance of CY15 Q3 LTO/LTM Report	35 days	Mon 8/31/15	Fri 10/16/15
317	SS067 - Conduct Monitoring, Prep and Submit CY15 Q4 LTO/LTM Report	20 days	Thu 10/1/15	Wed 10/28/15
318	SS067 - AFCEC & NYSDEC Review and Acceptance of CY15 Q4 LTO/LTM Report	35 days	Thu 10/29/15	Wed 12/16/15
319	**SS067 - Optimization Exit Strategy**	120 days	Wed 7/1/15	Tue 12/15/15
320	SS067 - Prepare and Submit Optimized Exit Strategy Report	45 days	Wed 7/1/15	Tue 9/1/15
321	SS067 - AFCEC & NYSDEC Review and Acceptance of Optimized Exit Strategy Report	75 days	Wed 9/2/15	Tue 12/15/15
322	**SS068 - BLDG 7001 (Closure Unrestricted Reuse) Closure Approval 12/8/14.**	**1033 days**	**Wed 4/27/11**	**Fri 4/10/15**
323	SS068 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11

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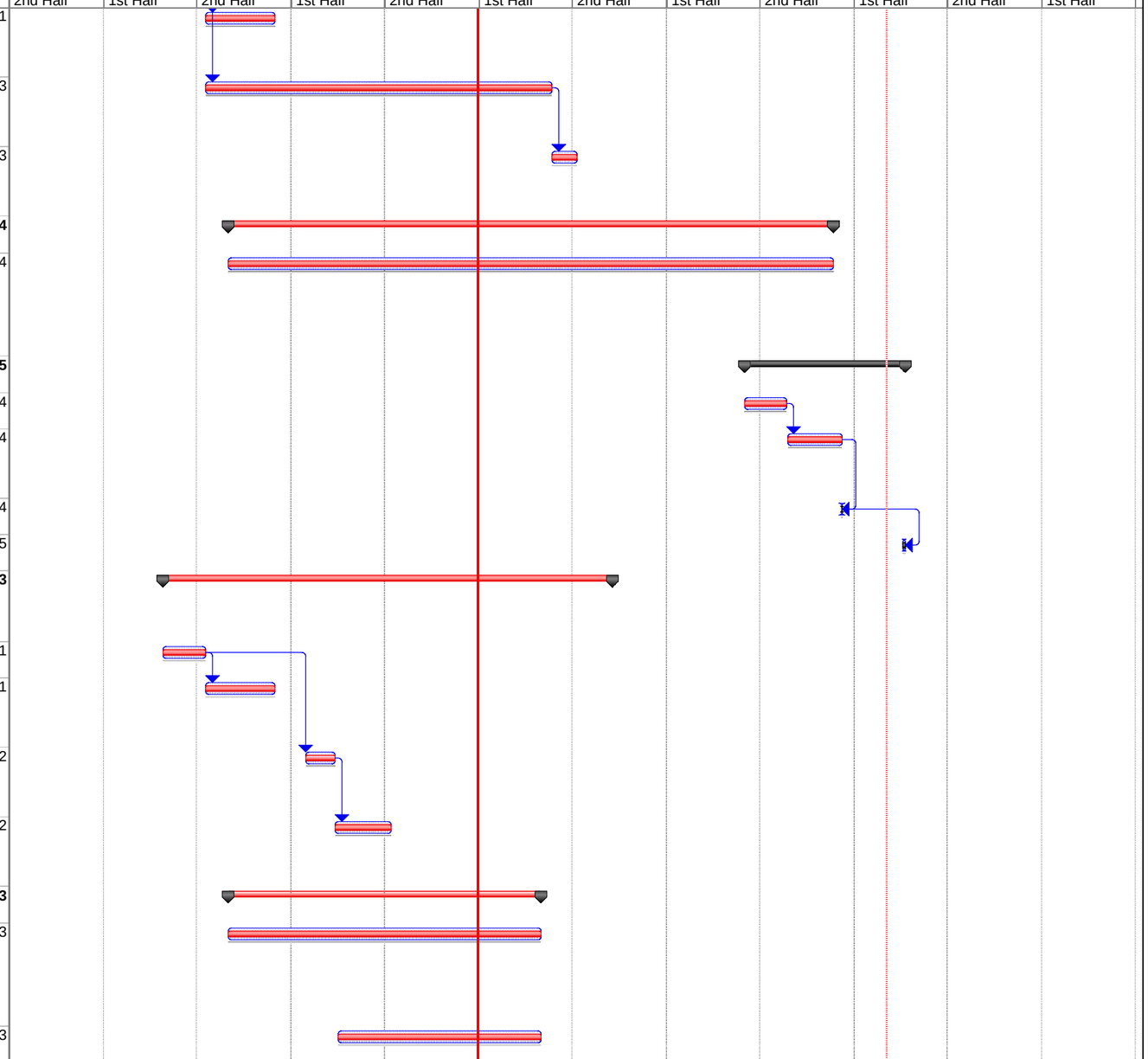
324	SS068 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
325	SS068 - Implement Remedy Optimization Systems and Submit Report	482 days	Tue 7/19/11	Wed 5/22/13
326	SS068 - AFCEC& NYSDEC Review and Acceptance of Optimization System and Report	35 days	Thu 5/23/13	Wed 7/10/13
327	**SS068 - Conduct Monitoring, Operations & Maintenance**	**842 days**	**Thu 9/1/11**	**Fri 11/21/14**
328	SS068 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports 2011, 2012, 2013 and 2014. Achieve AFCEC and NYSDEC Approval.	842 days	Thu 9/1/11	Fri 11/21/14
329	**SS068 - Achieve Site Closure**	**225 days**	**Mon 6/2/14**	**Fri 4/10/15**
330	SS068 - Prepare and Submit Closure Report	60 days	Mon 6/2/14	Fri 8/22/14
331	SS068 - AFCEC & NYSDEC Review and Acceptance of Closure Report	76 days	Mon 8/25/14	Mon 12/8/14
332	SS068 - NYSDEC Closure of Spill Number	1 day	Mon 12/8/14	Mon 12/8/14
333	SS068 - Wells Decommissioned	5 days	Mon 4/6/15	Fri 4/10/15
334	**SS069 - Bulk Fuel Storage Area (Closure Unrestricted Reuse) Closure Approval 5/1/13**	**625 days**	**Wed 4/27/11**	**Tue 9/17/13**
335	SS069 - Prepare and Submit Design/plan remedy optimization	59 days	Wed 4/27/11	Mon 7/18/11
336	SS069 - AFCEC & NYSDEC Review and Acceptance of Design/Plan Remedy Optimization	97 days	Tue 7/19/11	Wed 11/30/11
337	SS069 - Implement Remedy Optimization Systems and Submit Report	41 days	Mon 1/30/12	Mon 3/26/12
338	SS069 - AFCEC & NYSDEC Review and Acceptance of Optimization System and Report	79 days	Tue 3/27/12	Fri 7/13/12
339	**SS069 - Conduct Monitoring, Operations & Maintenance**	**435 days**	**Thu 9/1/11**	**Wed 5/1/13**
340	SS068 - Conduct Monitoring, Prep and Submit Quarterly LTO/LTM Reports 2011, 2012, and 2013. Achieve AFCEC and NYSDEC Approval.	435 days	Thu 9/1/11	Wed 5/1/13
341	**SS069 - Achieve Site Closure**	283 days	Mon 4/2/12	Wed 5/1/13



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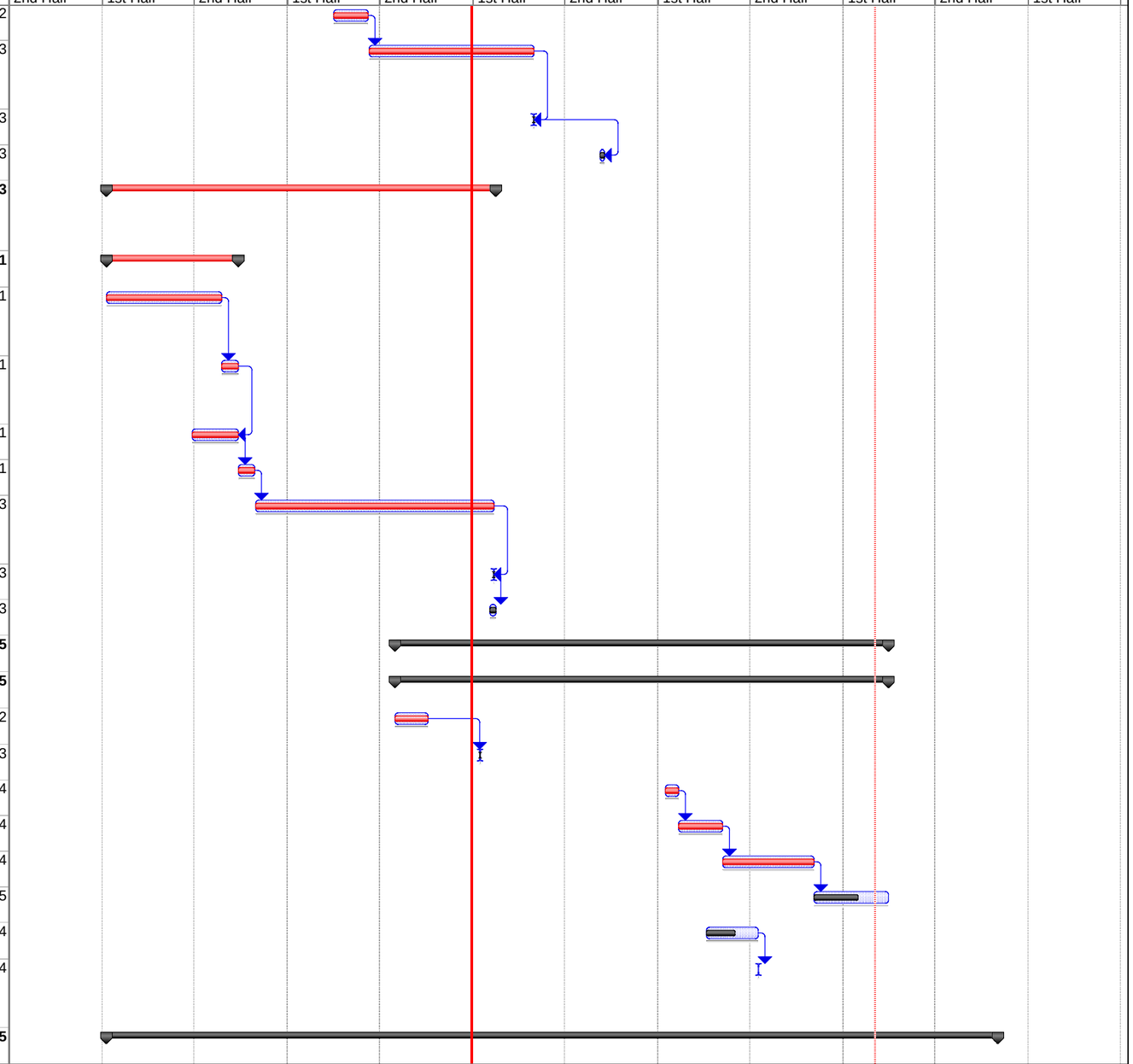
342	SS069 - Prepare and Submit Closure Report	50 days	Mon 4/2/12	Fri 6/8/12
343	SS069 - AFCEC & NYSDEC Review and Acceptance of Closure Report	233 days	Mon 6/11/12	Wed 5/1/13
344	SS069 - NYSDEC Closure of Spill Number	1 day	Wed 5/1/13	Wed 5/1/13
345	SS069 - Wells Decommissioned	7 days	Mon 9/9/13	Tue 9/17/13
346	**SS070 - Building 150 (Closure Unrestricted Use) Closure Approval 2/11/13**	**550 days**	**Mon 1/10/11**	**Fri 2/15/13**
347	**SS070 - Conduct Monitoring**	**186 days**	**Mon 1/10/11**	**Mon 9/26/11**
348	SS070 - Conduct Monitoring, Prep and Submit CY11 LTM Report	163 days	Mon 1/10/11	Wed 8/24/11
349	SS070 - AFCEC & NYSDEC Review and Acceptance of CY11 LTM Report	23 days	Thu 8/25/11	Mon 9/26/11
350	SS070 - Achieve Site Closure	65 days	Tue 6/28/11	Mon 9/26/11
351	SS070 - Prepare and Submit Closure Report	24 days	Tue 9/27/11	Fri 10/28/11
352	SS070 - AFCEC & NYSDEC Review and Acceptance of Closure Report	336 days	Mon 10/31/11	Mon 2/11/13
353	SS070 - NYSDEC Closure of Spill Number	1 day	Mon 2/11/13	Mon 2/11/13
354	SS070 - Wells Decommissioned	10 days	Mon 2/4/13	Fri 2/15/13
355	**Unknown Sites**	**695 days**	**Wed 8/1/12**	**Tue 3/31/15**
356	**Investigate 10 Unknown Sites**	**695 days**	**Wed 8/1/12**	**Tue 3/31/15**
357	**Investigation Report - Site 1: AOI-474**	65 edays	Wed 8/1/12	Fri 10/5/12
358	Acceptance of Investigation Report - Site 1:AOI 474	1 day	Tue 1/15/13	Tue 1/15/13
359	Draft Site Closure Report	18 days	Thu 1/16/14	Mon 2/10/14
360	NYSDEC Review (Request for additional samples)	63 days	Tue 2/11/14	Thu 5/8/14
361	Final Closure Report (Closure Pending)	128 days	Fri 5/9/14	Tue 11/4/14
362	NYSDEC & EPA Review and Acceptance of Closure Report	105 days	Wed 11/5/14	Tue 3/31/15
363	**Investigation Report - Site 2: Building 785 Pipeline**	74 days	Mon 4/7/14	Thu 7/17/14
364	Acceptance of Investigation Report - Site 2: Building 785 Pipeline	1 day	Fri 7/18/14	Fri 7/18/14
365	**Five-Year Review**	**1256 days**	**Mon 1/10/11**	**Mon 11/2/15**



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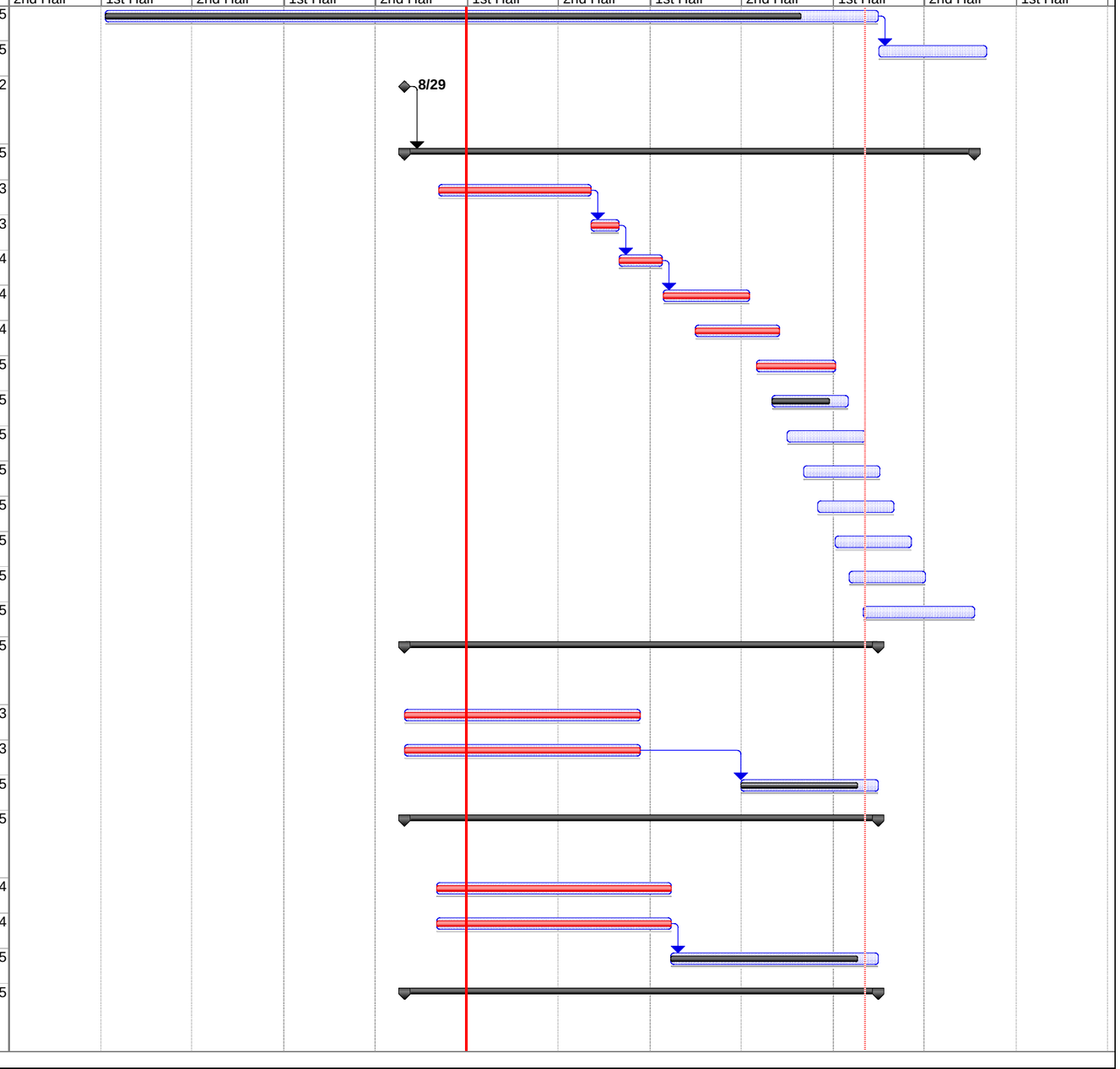
ID	Task Name	Duration	Start	Finish
366	Five-Year Review Report Submittal to NYSDEC and EPA	1102 days	Mon 1/10/11	Tue 3/31/15
367	Acceptance of Five-Year Review Report	153 days	Thu 4/2/15	Mon 11/2/15
368	**Contract Modification 002: Revised Site Objectives and New Sites (Seven revised objectives and two new sites)**	0 days	Wed 8/29/12	Wed 8/29/12
369	**ST006-Building 101 Unrestricted Site Closure**	813 days	Wed 8/29/12	Fri 10/9/15
370	Conduct SVI Evaluation	218 days	Mon 11/5/12	Wed 9/4/13
371	Option 2 System Installation and Prepare Operation Plan	40 days	Thu 9/5/13	Wed 10/30/13
372	Operation Plan and Start Up Draft Report	62 days	Thu 10/31/13	Fri 1/24/14
373	Operation Plan and Start Up Final Report	124 days	Mon 1/27/14	Thu 7/17/14
374	SVE System Start Up and O&M Quarter 1 Report	120 days	Tue 4/1/14	Mon 9/15/14
375	SVE System O&M Quarter 2 Report	112 days	Fri 8/1/14	Mon 1/5/15
376	SVE System O&M Quarter 3 Report	110 days	Mon 9/1/14	Fri 1/30/15
377	SVE System O&M Quarter 4 Report	112 days	Wed 10/1/14	Thu 3/5/15
378	SVE System O&M Quarter 5 Report	110 days	Mon 11/3/14	Fri 4/3/15
379	SVE System O&M Quarter 6 Report	110 days	Mon 12/1/14	Fri 5/1/15
380	SVE System O&M Quarter 7 Report	110 days	Mon 1/5/15	Fri 6/5/15
381	SVE System O&M Quarter 8 Report	110 days	Mon 2/2/15	Fri 7/3/15
382	SVE System Rebound Monitoring and Report / Site Closure Report	160 days	Mon 3/2/15	Fri 10/9/15
383	**DP012-Building 301 Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
384	Conduct Site Investigation Activities	336 days	Wed 8/29/12	Wed 12/11/13
385	Remedial Action Activities	336 days?	Wed 8/29/12	Wed 12/11/13
386	Site Closure Report	196 days	Tue 7/1/14	Tue 3/31/15
387	**DP013-Building 255 Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
388	Conduct Site Investigation Activities	334 days	Thu 11/1/12	Tue 2/11/14
389	Remedial Action Activities	334 days	Thu 11/1/12	Tue 2/11/14
390	Site Closure Report	296 days	Tue 2/11/14	Tue 3/31/15
391	**DP015-Building 219 Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15



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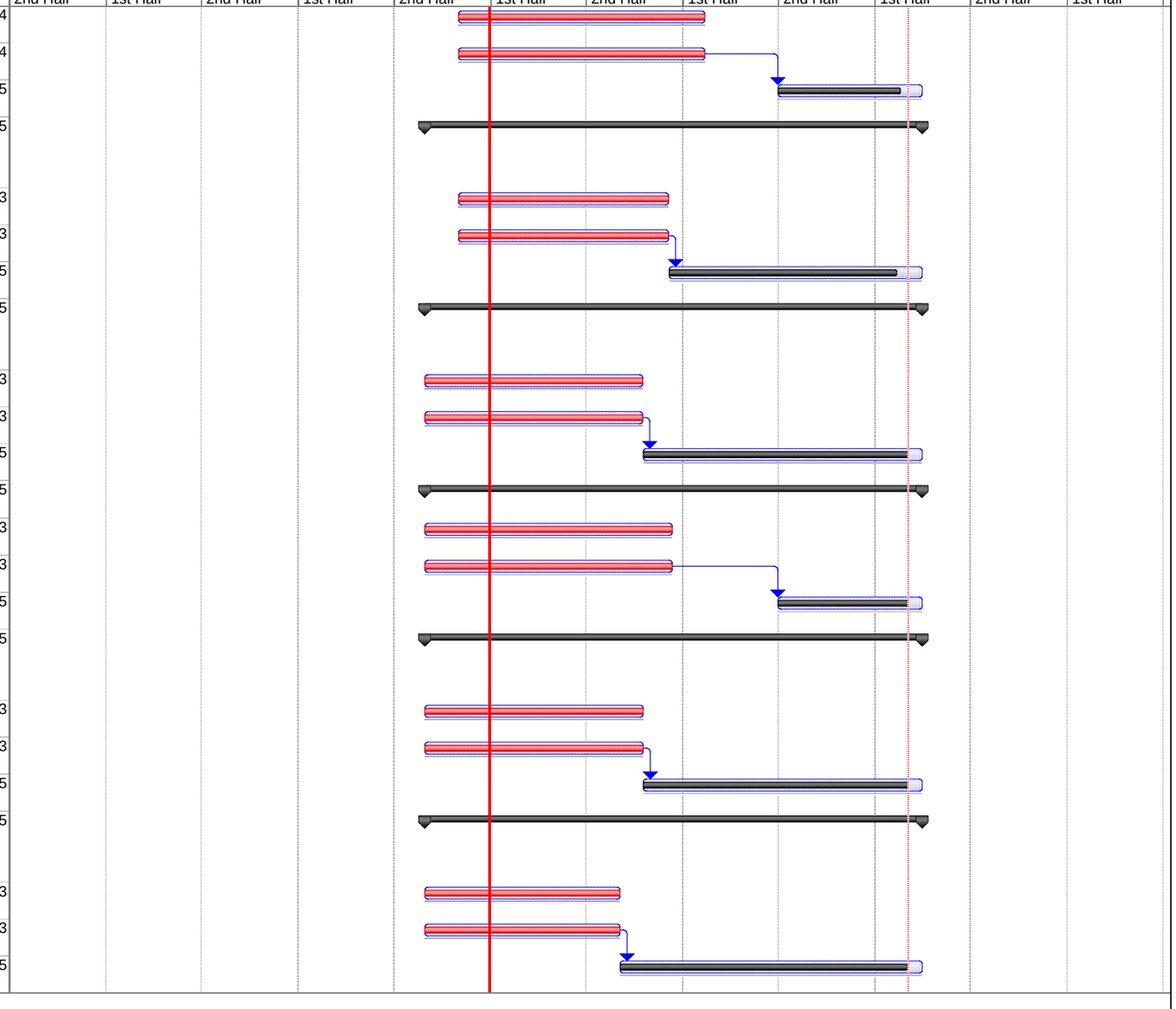
392	Conduct Site Investigation Activities	334 days	Thu 11/1/12	Tue 2/11/14
393	Remedial Action Activities	334 days	Thu 11/1/12	Tue 2/11/14
394	Site Closure Report	196 days	Tue 7/1/14	Tue 3/31/15
395	**SS024-Fire Demonstration Area Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
396	Conduct Site Investigation Activities	285 days	Thu 11/1/12	Wed 12/4/13
397	Remedial Action Activities	285 days	Thu 11/1/12	Wed 12/4/13
398	Site Closure Report	343 days	Fri 12/6/13	Tue 3/31/15
399	**SD050-Building 214 Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
400	Conduct Site Investigation Activities	296 days	Wed 8/29/12	Wed 10/16/13
401	Remedial Action Activities	296 days	Wed 8/29/12	Wed 10/16/13
402	Site Closure Report	378 days	Fri 10/18/13	Tue 3/31/15
403	**AOI 72 Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
404	Conduct Site Investigation Activities	336 days	Wed 8/29/12	Wed 12/11/13
405	Remedial Action Activities	336 days?	Wed 8/29/12	Wed 12/11/13
406	Site Closure Report	196 days	Tue 7/1/14	Tue 3/31/15
407	**Building 211 Drywell Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
408	Conduct Site Investigation Activities	297 days	Wed 8/29/12	Thu 10/17/13
409	Remedial Action Activities	297 days	Wed 8/29/12	Thu 10/17/13
410	Site Closure Report	378 days	Fri 10/18/13	Tue 3/31/15
411	**FT030- Fire Training Area Unrestricted Site Closure (Site Closure Pending)**	675 days	Wed 8/29/12	Tue 3/31/15
412	Conduct Site Investigation Activities	265 days	Wed 8/29/12	Tue 9/3/13
413	Remedial Action Activities	265 days	Wed 8/29/12	Tue 9/3/13
414	Site Closure Report	410 days	Wed 9/4/13	Tue 3/31/15



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