National Aeronautics and Space Administration

Final

Sampling and Analysis Plan

Operable Unit 3
Waste Oil Dump
Long Term Monitoring Plan
Revision 4

Goddard Space Flight Center
Wallops Flight Facility
Wallops Island, Virginia

August 2022

www.nasa.gov

CERTIFICATION

I certify that the information contained in or accompanying this document is true, accurate, and complete.

As to any portion of this document for which I cannot personally verify its accuracy, I certify under penalty of law that this document and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

DAVID LIU

Digitally signed by DAVID LIU
Date: 2022.08.30
09:35:52 -04'00'

Signature: ______________________________

Name: David Liu

Title: NASA Project Coordinator

Executive Summary

NASA Wallops Flight Facility (WFF) is updating the Long Term Monitoring (LTM) Plan for Operable Unit 3 Waste Oil Dump (WOD), located at the NASA Goddard Space Flight Center's WFF in Wallops Island, Virginia. The previous LTM Plan – Revision 3 included the collection and analysis from seven monitoring wells. Four of these monitoring wells have previously attained the cleanup goal established in the Record of Decision. This LTM plan includes the collection of groundwater samples for analysis of arsenic from three existing monitoring wells and synoptic water level measurements from these and nine additional monitoring wells.

This SAP outlines the project management, planned activities, data acquisition, oversight, and data review procedures necessary for the WOD groundwater LTM Plan. Based on human health and ecological risk assessments and exceedances of regulatory standards, the only analytical parameter included in the WOD groundwater LTM program is currently arsenic. The sole medium of concern at the WOD is shallow groundwater in the upper, unconfined zone of the Columbia aquifer; past disposal operations at the WOD are the likely source of groundwater contamination.

The objective of the LTM program for the WOD is to document the concentrations of total and dissolved arsenic in site groundwater, confirm groundwater flow, and monitor progress in attaining the clean-up goals established in the Record of Decision. Field and analytical activities will be conducted in accordance with the Standard Operating Procedures identified in the SAP.

TABLE OF CONTENTS **PAGE**

TABLE

FIGURES

APPENDICES

List of Abbreviations and Acronyms

%R	percent recovery
°C	degrees Celsius
µg/L	micrograms per liter
AAOC	Administrative Agreement on Consent
APP	Accident Prevention Plan
Bluestone	Bluestone Environmental Group, Inc.
CA	corrective action
COC	Contaminant of Concern
DL	detection limit
DO	dissolved oxygen
DQI	data quality indicator
DQO	data quality objective
EDD	electronic data deliverable
EPA	Environmental Protection Agency
FCA	Former Circular Area
FD	Field Duplicate
FS	Feasibility Study
FSM	Field Site Manager
GAC	granular activated carbon
GW	groundwater
HASP	Health and Safety Plan
H&S	health and safety
HAZWOPER	Hazardous Waste Operations and Emergency Response
ICP/MS	inductively coupled plasma/mass spectrometry
IDQTF	Intergovernmental Data Quality Task Force
IDW	investigation-derived waste
LCS	laboratory control sample
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
mL/min	milliliters per minute
MPC	method performance criteria
MS	matrix spike
MSD	matrix spike duplicate
mV	millivolts
MW	monitoring well
NASA	National Aeronautics and Space Administration
NTUs	Nephelometric Turbidity Unit
ORP	oxidation-reduction potential
ORC	Oxygen Releasing Compound
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAL	project action limits
PDF	portable document format
PG	Professional Geologist
pH	hydrogen ion activity
PID	photoionization detector
PM	Project Manager
POC	Point of Contact
PPE	personal protective equipment
PQLG	project quality limit goal

PRAP	Proposed Remedial Action Plan
QA	quality assurance
QAPP	Quality Assurance Project Plan
QAM	Quality Assurance Manager
QC	quality control
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
ROD	Record of Decision
RPD	relative percent difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SI	Site Investigation
SOP	Standard Operating Procedures
SSHO	Site Safety and Health Officer
TtNUS	Tetra Tech NUS, Inc
U. S.	United States
VDEQ	Virginia Department of Environmental Quality
VELAP	Virginia Environmental Laboratory Accreditation Program
WFF	Wallops Flight Facility
WOD	Waste Oil Dump
WWTP	Wastewater Treatment Plant

SAP Worksheet #1: Title and Approval Page

(UFP-QAPP Manual Section 2.1- Worksheet #1)

Document Title: Quality Assurance Project Plan (SAP), Operable Unit 3, Waste Oil Dump, Long Term Monitoring Plan – Revision 4, Goddard Space Flight Center, Wallops Flight Facility (WFF), Wallops Island, Virginia

Lead Organization: National Aeronautics and Space Administration (NASA)

Preparer's Address, Telephone Number, and email Address: 301 Lindenwood Drive, Malvern, Pennsylvania, 19355, 610-647-9500, harry.m.stein@nasa.gov

Preparation Date (Day/Month/Year): June 2, 2022

This SAP and Long Term Monitoring Plan was prepared and is submitted in accordance with the requirements of the 2004 Resource Conservation and Recovery Act (RCRA) Administrative Agreement on Consent (AAOC) between NASA and the United States (U.S.) Environmental Protection Agency (EPA), EPA Docket Number RCRA-03-2004-0201TH.

Investigative Organization's Project Manager (PM):

Marianne Simko (affiliate)
Digitally signed by Marianne Simko (affiliate)
Date: 2022.08.29 15:02:24 -04'00'

Signature
Marianne Simko
Bluestone PM

Date

Investigative Organization's Project Quality Assurance (QA) Manager (QAM):

8/24/2022

Signature
Virginia Pohlman
Bluestone QAM

Date

Lead Organization's Remedial Project Manager (RPM):

DAVID LIU
Digitally signed by DAVID LIU
Date: 2022.08.30 08:57:24 -04'00'

Signature
David Liu
NASA RPM

Date

SAP Worksheet #2: Distribution List

(UFP-QAPP Manual Section 2.3.1-Worksheet #3)

SAP Recipient	Project Title/Role	Organization	Telephone Number	Email Address
David Liu	NASA RPM	NASA	757-824-2141	David.Liu-1@nasa.gov
Harry Stein	Program Manager	Bluestone Environmental Group, Inc.	443-864-7490	harry.m.stein@nasa.gov
Marianne Simko	Deputy Program Manager/PM	Bluestone Environmental Group, Inc.	757-710-5904	marianne.f.simko@nasa.gov
Susan Dunn	Facility POC	Bluestone Environmental Group, Inc.	757-824-1832	Susan.K.Dunn@nasa.gov
Andy Johnson	Laboratory PM	Eurofins TestAmerica Pittsburgh	615-818-9567	Andy.Johnson@Eurofinset.com
Virginia Pohlman	QAM	Bluestone Environmental Group, Inc.	484-883-1657	gpohlman@bluestoneenviro.com
Florence Sevold	Data Manager	Bluestone Environmental Group, Inc.	610-647-9500 x1019	fsevold@bluestoneenviro.com
Emily Strake	Data Validation Manager	Bluestone Environmental Group, Inc.	267-300-6309	estrake@bluestoneenviro.com
Kyle Newman	VDEQ RPM	Virginia Department of Environmental Quality	804-659-1322	kyle.newman@deq.virginia.gov
Lorie Baker	EPA RPM	Environmental Protection Agency	215-814-3355	baker.lorie@epa.gov
Skylar Georgius	SSHO / Field Geologist	Bluestone Environmental Group, Inc.	301-366-9519	sgeorgius@bluestoneenviro.com

SAP Worksheet #3 Personnel Qualifications and Sign-off Sheet

(UFP-QAPP Manual Section 2.3.2- Worksheet #4)

ORGANIZATIONS: **National Aeronautics and Space Administration (NASA), Bluestone Environmental Group, Inc. (Bluestone)**

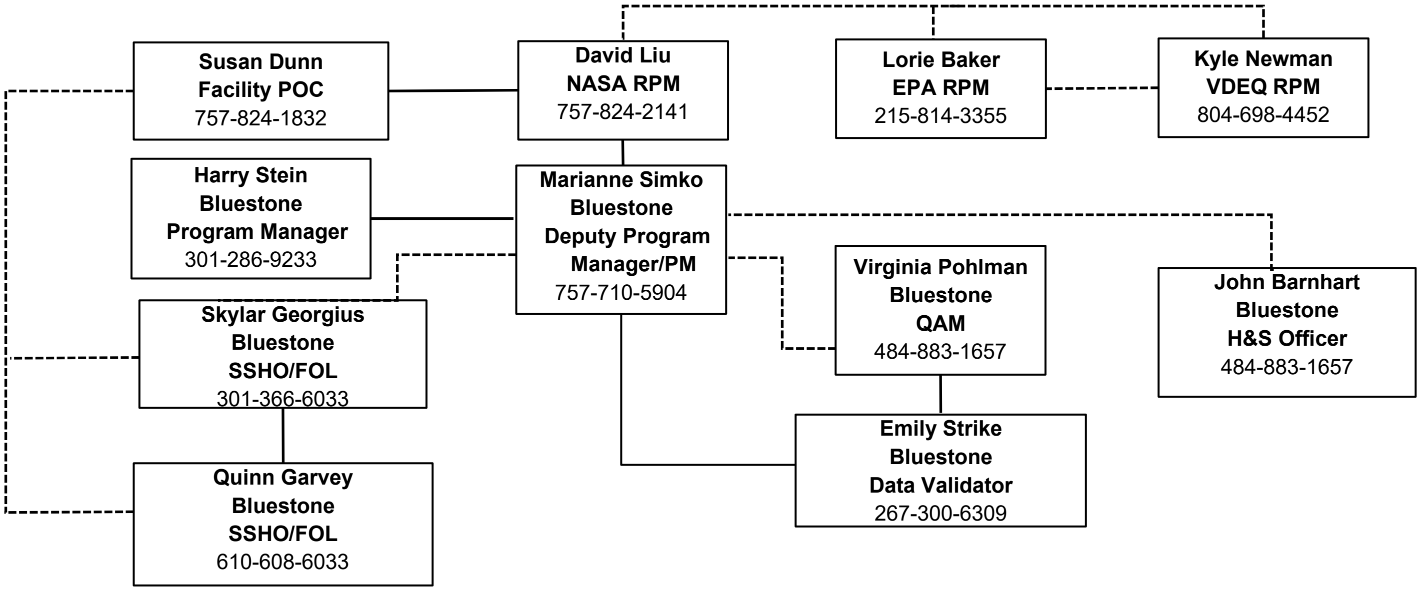
Name	Organization/Title	Telephone Number	Signature	Date SAP Read
David Liu	NASA RPM	757-824-2141		
Marianne Simko	Bluestone Deputy Program Manager/PM	757-710-5904		
Susan Dunn	NASA/Bluestone Facility Point of Contact (POC)	410-430-3060		
Virginia Pohlman, PE	Bluestone QAM	484-883-1657		
Skylar Georgius, PG	Bluestone Field Geologist/Site Safety and Health Officer	301-366-9519		
Quinn Garvey	Bluestone Alternate Site Safety and Health Officer	610-608-6033		
Emily Strake	Data Validator	267-300-6309		

ORGANIZATION: **Eurofins Test America Pittsburgh Laboratories Environmental, LLC (Laboratory)**

Name	Organization/Title	Telephone Number	Signature	Date SAP Read
Andy Johnson	Client Service Manager	615-818-9567	N/A	N/A

SAP Worksheet #4 Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1- Worksheet #5)



Lines of Authority ————————

Lines of Communication ----------------------------------

SAP Worksheet #5: Communication Pathways

(UFP-QAPP Manual Section 2.4.2-Worksheet #6)

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory Interface	NASA	David Liu	757-824-2141	The NASA PM will be the direct liaison with EPA and VDEQ.
Regulatory oversight/ involvement	EPA VDEQ	Lorie Baker Kyle Newman	215-814-3355 804-659-1322	Responsible for regulatory approval. Will receive status updates from NASA.
SAP and Work Plan Amendments	Bluestone NASA	Marianne Simko David Liu	757-710-5904 757-824-2141	The NASA PM will initiate changes to the SAP & Work Plan, which will be implemented by the Project Team (e.g., additional sample locations, additional analytical suites, etc.). NASA must approve all changes prior to implementation. Changes will be documented via amendments and copies will be forwarded to all parties.
Changes to field work	Bluestone	Marianne Simko	757-710-5904	The NASA PM will be notified within 24 hours. Telephone and e-mail notifications are acceptable. All field changes will be documented.
Daily field activities reporting	Bluestone	Marianne Simko	757-710-5904	The PM will e-mail field documentation to the NASA PM daily.
Field corrective action	NASA PM	David Liu	757-824-2141	The PM determines the need for corrective actions. Corrective actions may also be identified by the field team.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
	Bluestone	Skylar Georgius	301-366-9519	The PM determines the need for corrective actions. Corrective actions may also be identified by the field team. Field Site Manager (FSM) initiates corrective action on identified field issues immediately or within recommended timeframe.
Review of Data Validation	Bluestone	Emily Strake	267-300-6309	Data quality objective (DQO) development, laboratory coordination and oversight. Management of analytical lab/analytical Cas/release of analytical data. Data validation issues and data review corrective actions. Address laboratory quality control and programmatic analytical data questions.
Reporting laboratory data quality issues	Eurofins Test-America Pittsburgh	Andy Johnson	Andy.Johnson@Eurofinset.com	All QA/QC issues with project field samples will be reported to Bluestone and the NASA Project Chemist as soon as possible, and no longer than within 2 business days.
Health and safety (H&S)	Bluestone	John Barnhart	301-606-8996	Implement and provide oversight for H&S program. Coordinate with PM for any changes, issues, or H&S incidents.
Safety decisions; reporting of safety issues; tailgate safety meeting	Bluestone	Skylar Georgius Or Quinn Garvey and Field Technicians	301-366-9519 610-608-6033	Implement Health and Safety Plan; recommend changes due to changes in field conditions. Inform NASA PM of any changes. Hold safety briefings. All project team members share an obligation to immediately stop work if a potential unsafe act is being observed and requires corrective action.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Project Report Review	Bluestone Bluestone NASA	Susan Dunn Marianne Simko David Liu	757-824-1832 757-710-5904 757-824-2141	Internal reviews will be conducted by the Bluestone PM and support staff. Comments will be resolved internally to the satisfaction of the Bluestone PM. When satisfied with resolution of all comments, the report will be submitted by the Bluestone PM or designee to the NASA PM in accordance with the project scope of work requirements. Upon resolution of NASA comments, the report will be submitted to the NASA PM for transmittal to the regulators for review. Comments will be resolved to the satisfaction of the NASA PM, revisions will be made, and the report will be finalized for final distribution and submittal.

SAP Worksheet #6: Personnel Responsibilities and Qualifications

(UFP-QAPP Manual Section 2.4.3- Worksheet #7)

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
David Liu	Remedial Project Manager (RPM)	NASA	Oversees implementation of the AAOC at NASA WFF.	To be provided upon request
Lorie Baker	EPA RPM	EPA	Oversees project management on behalf of the EPA	To be provided upon request
Kyle Newman	VDEQ RPM	VA DEQ	Oversees project management on behalf of the Virginia Department of Environmental Quality.	To be provided upon request
Aaron Myers	Vice President	Bluestone	Contractual responsibilities.	To be provided upon request
Marianne Simko	Deputy Program Manager/PM	Bluestone	Overall project quality, schedule and management; Project management and administrative activities. Ensures health and safety requirements are implemented. Oversees project financial, schedule, and technical management.	BS, Civil Engineering 24 years of experience
Virginia Pohlman, PE	QC Supervisor	Bluestone	Ensures documents and collected data meet quality standards.	MS, Civil Engineering/BS, Civil Engineering 24 years of experience
John Barnhart	Health and Safety (H&S)	Bluestone	Implements and provides oversight for H&S program. Coordinates with PM for any changes, issues, or H&S incidents.	MS, Occupational Safety and Health/ 42 years of experience
Skylar Georgius, PG	Field Geologist/Site Safety and Health Officer	Bluestone	Supervises, coordinates and performs field sampling activities. Ensures health and safety protocols are followed. Functions as on site communication to management. Oversees mobilization and demobilization of all field equipment and subcontractors.	BA, Geology 6 years of experience

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
Quinn Garvey	Alternate Site Safety and Health Officer	Bluestone	If necessary, the alternate supervises, coordinates, and performs field sampling activities. Ensures health and safety protocols are followed. Functions as on site communication to management. Oversees mobilization and demobilization of all field equipment and subcontractors.	BS, Environmental Systems Engineering 3 years of experience
Susan Dunn	Facility POC	Bluestone	Coordinates site access and assists with field sampling activities.	BS, Biology 19 years of experience
Emily Strake	Data Validator	Bluestone	Validates analytical data.	BS, Chemistry/MBA 22 years of experience
Andy Johnson	Laboratory PM	Eurofins Test - America Pittsburgh	Project management on behalf of the laboratory.	To be provided upon request

SAP Worksheet #7: Special Personnel Training Requirements

(UFP-QAPP Manual Section 2.4.4- Worksheet #8)

Project specific safety and training requirements are presented in the contract wide approved Health and Safety Plan. Sampling technicians will be required to have Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) certification.

SAP Worksheet #8: Project Scoping Session

(UFP-QAPP Manual Section 2.5.1- Worksheet #9)

Project scoping sessions that apply to the Waste Oil Dump Long Term Monitoring Program will be held with regulators and stakeholders on an as-needed basis. Scoping sessions will be documented in the template below.

Project Name:	WOD LTM Program	**Site Name:**	WOD
Projected Date(s) of Sampling:	TBD	**Site Location:**	WFF
Project Manager:	David Liu (NASA); TBD		
Date of Session:	TBD		
Scoping Session Purpose:	Project kick-off call		

Name	**Title**	**Affiliation**	**Phone #**	**Email Address**
David Liu	NASA RPM	NASA	757-824-2141	David.Liu-1@nasa.gov
Susan Dunn	Facility POC and Fieldwork Coordination	Bluestone Environmental Group Inc.	757-824-1832	Susan.k.dunn@nasa.gov
TBD	PM	TBD	TBD	TBD
TBD	TBD	TBD	TBD	TBD

Comments: TBD

Action Items: TBD

SAP Worksheet #9: Problem Definition

(UFP-QAPP Manual Section 2.5.2- Worksheet #9)

9.1 PHYSICAL SETTING

WFF is located in Accomack County, Virginia and is comprised of three land parcels including the Main Base, Mainland, and Wallops Island (Figure 9-1). The Main Base consists of approximately 2,000 acres and is located near Virginia Routes 175 and 798. The Mainland is located six miles south of the Main Base on Virginia Route 679 and consists of approximately 1,207 acres, the majority of which is wetlands. Wallops Island, which is approximately 3,395 acres is a 7-mile-long barrier island which is connected to the Mainland by a causeway.

The majority of the WFF Main Base is located on a high terrace landform (25 to 40 ft above mean sea level [msl]) with the northern and eastern portions located on low terrace (0 to 25 ft msl) and tidal marsh (NASA WFF, 2018). The runways, which include Runway 10-28, Runway 17-35, and Runway 04-22, cover a large portion of the Main Base and form a flat plateau-like feature covering most of the higher elevations above msl. The Town of Chincoteague is located approximately 5 miles east of the Main Base.

The WOD is an open area located on the north side of the WFF Main Base at the northern end of Runway 17-35 (Figure 9-2) and was used for disposal of waste oils and possibly solvents from the 1940s through the 1950s. No records are available that detail the types and quantities of materials disposed at the Site. A review of aerial photographs from 1943 through 1994 indicated the presence of ground scarring and possible excavation at the former Waste Oil Dump from 1943 to 1961 (Tetra Tech, 2015). The northern, eastern, and western boundaries of the site are steeply sloped with surface water runoff into the marshes that border an unnamed tributary of Little Mosquito Creek.

The WOD is bordered to the west and southwest by two Formerly Used Defense Sites, Operable Unit 4 - Abandoned Drum Dump, and Operable Unit 5 - Site 15 Debris Pile. These sites are managed under a separate AAOC between NASA and the EPA.

9.2 FACILITY HISTORY

NASA Goddard Space Flight Center (GSFC) manages WFF, the oldest active launch range in the continental U.S. and the only rocket testing and launch range owned and operated by NASA. NASA's predecessor, the National Advisory Committee for Aeronautics (NACA), established a rocket launch site on the southern portion of Wallops Island in 1945. From 1945 to 1957, the facility was known as the Pilotless Aircraft Research Station and served as a high-speed aeronautical launch site. NACA constructed launch, radar support, and experimental facilities and continued operations until NASA was established in 1958. The Pilotless Aircraft Research Station then became known as Wallops Station and became involved in the civilian space program. NASA expanded its presence with the acquisition of the Main Base and Mainland parcels in 1959, following the property transfer from the Department of Defense. Wallops Station was named Wallops Flight Center in 1974 and the name was changed to WFF in 1981/1982 when the facility became part of GSFC (NASA, 2018).

From 1942 until the property transfer to NASA in 1959, the Navy operated the Chincoteague Naval Auxiliary Air Station at the Main Base. The Navy constructed runways, buildings, and other support structures for

Department of Defense aviation and aviation ordnance testing and training. NASA continues to maintain the runways and still occupies many of the buildings that were present in 1959.

The mission of WFF has undergone several changes since first established, but the main focus has been aeronautical and rocket research, scientific balloons, management of suborbital projects, suborbital and orbital tracking, and space technology research. WFF also provides unique services to NASA, Department of Defense, academia, and the commercial space industry (NASA, 2018). NASA does not manufacture rockets or rocket fuels/propellants at WFF. Rocket motors are transported to WFF from other facilities.

9.3 GEOLOGICAL AND HYDROGEOLOGIC SETTING

The geology of the eastern shore of Virginia can be characterized as a series of layered, unconsolidated sediments, that comprise an eastward thickening wedge that dips to the northeast towards the Atlantic Ocean. Approximately, 7,000 feet of sediment lies atop crystalline basement rock on site. The two stratigraphic groups encountered at the site are the Chesapeake Group and the overlying Columbia Group. The geology of the site does not significantly differ from the known regional geology.

The Columbia Group lithologic unit underlies the Waste Oil Dump area. Surface soils consist of fine- to medium-grained quartz sand with some silt of the Columbia Group which is approximately 50-feet thick. A 20-40-foot thick clay and silt aquitard isolates the Columbia Group from the underlying Yorktown aquifer. A sandy clay layer as much as five feet thick is present at depths ranging from 10 to 27 feet below ground surface at the site (TtNUS, 2008).

The hydrogeology of the eastern shore of Virginia consists of four aquifers: the upper, middle, and lower Yorktown-Eastover aquifers and the surficial Columbia aquifer. The Columbia aquifer is an unconfined aquifer with the upper Yorktown-Eastover confining unit acting as its lower boundary. Below the Columbia aquifer is the Yorktown-Eastover aquifer system, a freshwater multi-aquifer unit consisting of a series of sandy layers separated by finer grained silt and clay layers of the Yorktown and Eastover Formations. The Yorktown-Eastover aquifers are typically subdivided into the upper, the middle, and the lower Yorktown-Eastover aquifers, each of which exist under confined or semi-confined conditions. Below the lower Yorktown-Eastover aquifer is the St. Mary’s confining unit. The St. Mary’s confining unit is predominantly clay with varying amounts of silt, sand, and shells. The hydrology of the site does not significantly differ from the known regional hydrology. Historical measurements show that regional groundwater flow in the area is generally to the north, discharging to Little Mosquito Creek. The near surface overburden aquifer is primarily in sand with some silt of the Columbia Group.

Groundwater within the study area is encountered at depths ranging from 6 to 23 ft bgs. The deeper elevations of groundwater correspond to higher topography in the southern portion of the Site. Groundwater is at or immediately below the ground surface within the marsh areas. Based on historical well gauging information collected during the LTM, there is a groundwater divide that trends to the north-northwest with flow towards the west and the northeast, discharging to Little Mosquito Creek and its adjacent marshes (Tetra Tech, 2015). Table 9-1 summarizes the monitoring well screened intervals and well depths.

9.4 SITE HISTORY

Historical disposal operations conducted on the facility resulted in releases of hazardous materials and impacts to environmental media. Multiple lines of evidence suggest that waste oil was disposed at the Site and previous investigations addressed various constituents of waste oil. These investigations indicate that residual arsenic is still present in groundwater at the Site.

Environmental actions began at the WOD in 1986 when the Commonwealth of Virginia conducted an inspection and identified the presence of waste (reported as waste oil) on the surface of the Site. NASA removed approximately 180 cubic yards of contaminated soil from four separate areas of the Site in November and December 1986. A Preliminary Assessment (PA) consisting of interviews, review of historical records, and a site visit was conducted in 1988. A SI was conducted from 1989 through 1992. Field activities included soil-gas surveys, monitoring well installation, and sampling and analysis of soil, groundwater, and sediment.

In 1997, a monitoring well was installed in the WOD and sampled as a background well for investigations being conducted at the adjacent Site 15. Significant contamination was found in this well and the results were used to plan further investigations.

A Remedial Investigation (RI) was conducted from 1998 through 2000 and included a review of historical data, a geophysical survey, the installation of temporary and permanent monitoring wells, and sampling and analysis of soil and groundwater.

A Supplemental RI field investigation was conducted in 2003 (TtNUS, 2004). Field activities included monitoring well installation and sampling and analysis of soil and groundwater.

A Feasibility Study (FS) (TtNUS, 2005) was conducted in 2005 in which the remedial action objectives and general response actions were discussed, along with a presentation of remedial technologies and alternatives.

Based on the results of the FS, a Proposed Remedial Action Plan (PRAP) (TtNUS, 2007) was prepared, presenting remedial alternatives for the site. The selected remedy was documented in the Record of Decision (TtNUS 2008). The ROD stated that a Pilot Study would be conducted prior to full implementation of the selected remedial strategy.

A Pilot Study was conducted from December 2008 through January 2009 (TtNUS, 2009). Field activities included the installation of monitoring well WOD MW008, injection of ORC, and the collection of groundwater samples. As a result of the Pilot Study, a recommendation was made to initiate a full-scale injection of ORC to address groundwater contamination in the target area.

The Remedial Action for groundwater was implemented at WOD beginning from November 2009 through March 2010 (TtNUS, 2011). Field activities included the full-scale injection of ORC followed by a 3-month post-injection groundwater sampling.

Following the completion of the Remedial Action, the groundwater LTM program was initiated with the first LTM sampling event conducted in June 2010. The objective of the LTM program was to document the reduction in concentrations of site groundwater Contaminants of Concern (COC), benzene and arsenic, to meet the clean-up goals identified in the ROD (TtNUS, 2009b). The clean-up goal concentrations for groundwater COCs were 5 micrograms/Liter (µg/L) for benzene and 10 µg/L for arsenic.

Subsequent long-term groundwater monitoring events conducted from 2010 to 2014 prompted the removal of benzene from the LTM program since its performance monitoring goal established in the Record of Decision (ROD) was met. Currently, only arsenic is included in the LTM Plan. The LTM program consisted of semi-annual sampling through October 2016. Beginning in 2017, the sampling frequency was reduced from semi-annual to twice every five years with one sampling event in the spring and one in the fall for seven monitoring wells. These wells include WOD-MW003R, 15-MW001, 15-MW002, 15-MW007, WOD-MW002S, WOD-MW002D, and WOD-MW008. The last sampling event was completed in July 2020 (LJT, 2021). Concentrations of arsenic have remained below the clean-up goal in monitoring wells 15-MW002, WOD-MW003R, and WOD-MW008 since the implementation of the LTM program. In addition, the performance monitoring goal established in the ROD for four consecutive monitoring events has been achieved in monitoring well WOD-002S, with the last detection above the cleanup goal in December 2010 (Table 9-2).

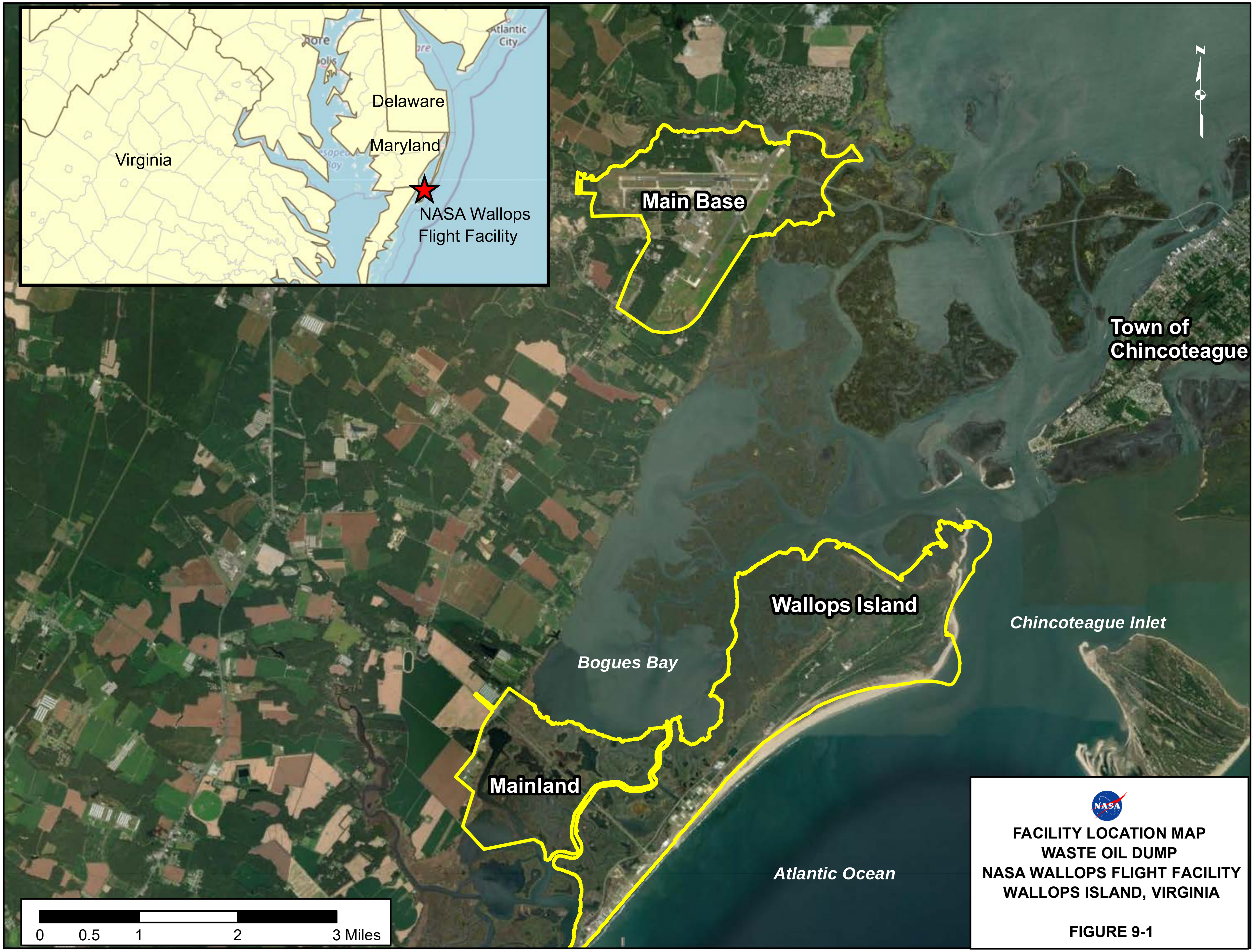
The previous LTM plan includes the collection of groundwater samples for analysis of arsenic from seven existing monitoring wells and synoptic water level measurements from these and five additional monitoring wells (Figure 9-3). NASA is recommending the removal of monitoring wells 15-MW002, WOD-002S, WOD-MW003R, and WOD-MW008 from the LTM program. Monitoring will continue at 15-MW001, 15-MW007, and WOD-MW002D

The known or suspected primary waste oil-related contaminants present in Site groundwater are listed below, as documented in the LTM Plan (Tetra Tech, 2015). There are no known co-occurring chemicals at the Site.

Primary Contaminants: Metals – arsenic
Cleanup goal for arsenic – 10 µg/L

Primary release mechanisms: Leaching to groundwater

During the July 2020 LTM event, samples were collected from six monitoring wells (the 7th well went dry during purging and did not recharge) and analyzed for total and dissolved arsenic. Total and dissolved arsenic was detected in samples from four of six monitoring wells with concentrations ranging from 5.4 µg/L to 59 µg/L. Total arsenic concentrations exceeded the site cleanup goal of 10 µg/L in two monitoring wells (Figure 9-4), (LJT, 2021). No changes to the LTM plan were recommended at the time. The performance monitoring goals established in the ROD have been achieved at monitoring wells 15-MW002, WOD-002S, WOD-MW003R, and WOD-MW008. The removal of these four wells from the LTM program starting with the September 2022 sampling event is recommended. PFAS investigations at WFF are ongoing. These wells will remain in place and will be properly maintained for future sampling, as needed.

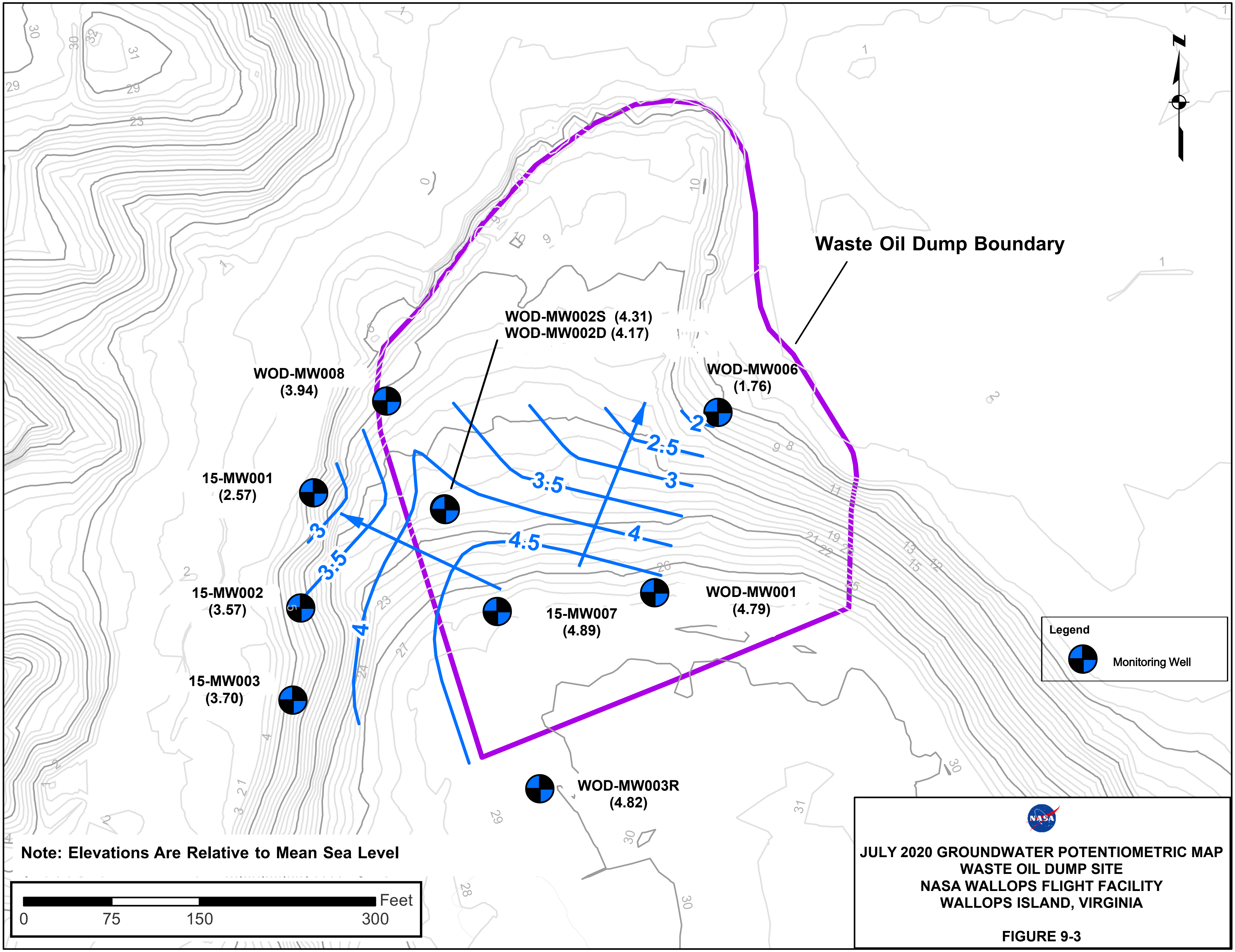


FACILITY LOCATION MAP
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA

FIGURE 9-1

SITE LOCATION MAP
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA

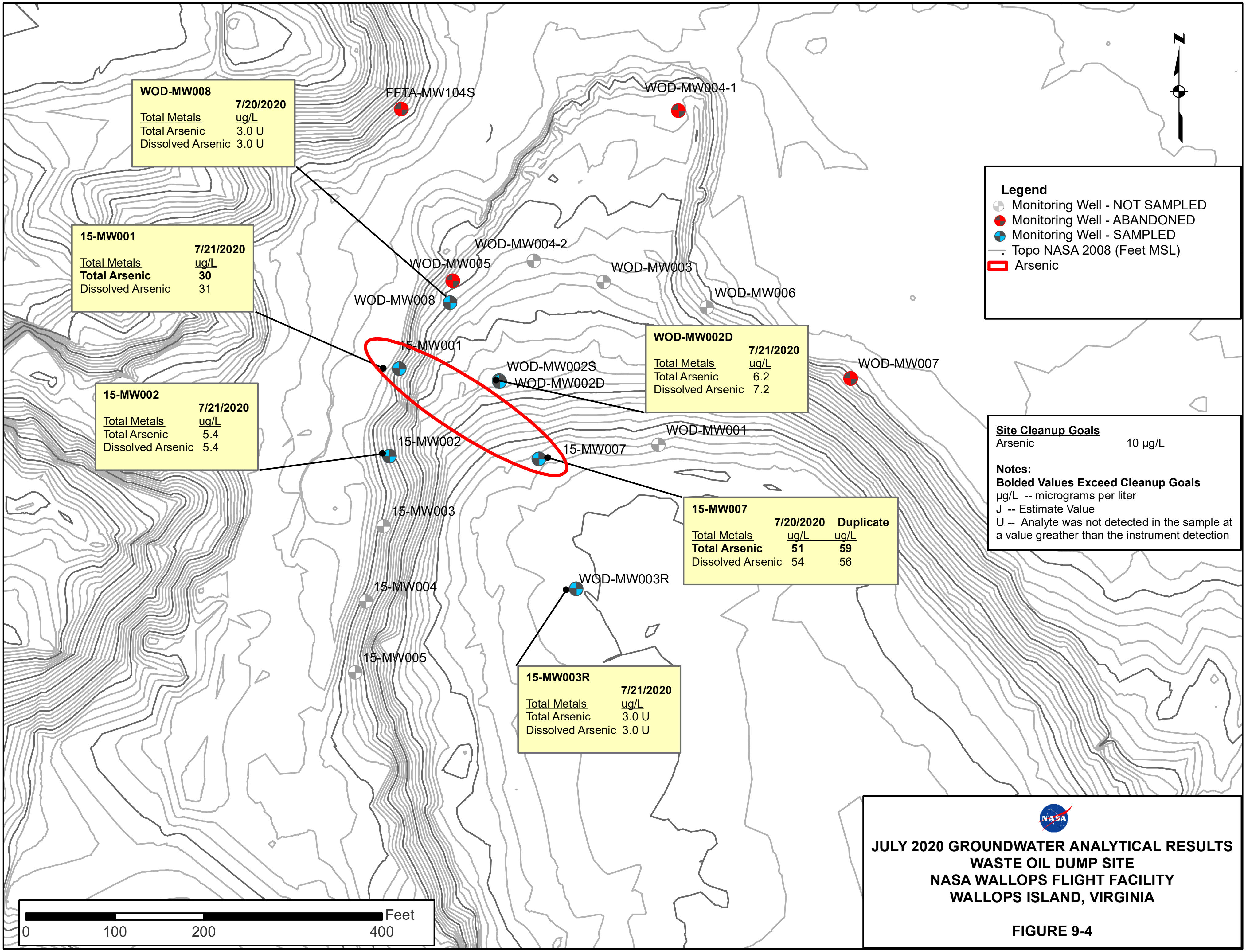
FIGURE 9-2



Note: Elevations Are Relative to Mean Sea Level

JULY 2020 GROUNDWATER POTENTIOMETRIC MAP
WASTE OIL DUMP SITE
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA

FIGURE 9-3



WOD-MW008

	7/20/2020
Total Metals	ug/L
Total Arsenic	3.0 U
Dissolved Arsenic	3.0 U

15-MW001

	7/21/2020
Total Metals	ug/L
Total Arsenic	**30**
Dissolved Arsenic	31

15-MW002

	7/21/2020
Total Metals	ug/L
Total Arsenic	5.4
Dissolved Arsenic	5.4

WOD-MW002D

	7/21/2020
Total Metals	ug/L
Total Arsenic	6.2
Dissolved Arsenic	7.2

15-MW007

	7/20/2020	Duplicate
Total Metals	ug/L	ug/L
Total Arsenic	**51**	**59**
Dissolved Arsenic	54	56

15-MW003R

	7/21/2020
Total Metals	ug/L
Total Arsenic	3.0 U
Dissolved Arsenic	3.0 U

Legend

- ◐ Monitoring Well - NOT SAMPLED
- 🟥 Monitoring Well - ABANDONED
- ◑ Monitoring Well - SAMPLED
- — Topo NASA 2008 (Feet MSL)
- ▭ Arsenic

Site Cleanup Goals

Arsenic	10 µg/L

Notes:

Bolded Values Exceed Cleanup Goals

µg/L -- micrograms per liter

J -- Estimate Value

U -- Analyte was not detected in the sample at a value greather than the instrument detection

JULY 2020 GROUNDWATER ANALYTICAL RESULTS
WASTE OIL DUMP SITE
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA

FIGURE 9-4

TABLE 9-1
GROUNDWATER MONITORING WELL SUMMARY
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA

Monitoring Well (actual)	Monitoring Well (alias)	Screened Interval (ft bgs)	Total Depth BTOC (ft)	Surveyed Elevation Top of Casing (ft)
WOD-MW003R	15-MW3R	21-31	32.71	32.42
15-MW001	15-GW1	1-5	10.19	6.27
15-MW002	15-GW2	1-5	9.02	9.21
15-MW003	15-GW3	1-5	10.20	8.70
15-MW007	15-GW7	15-30	32.96	30.58
WOD-MW001	16-GW1	17-22	26.22	30.39
WOD-MW002D	16-GW2D	23-28	32.07	21.35
WOD-MW002S	16-GW2S	9-19	23.01	21.19
WOD-MW003	16-GW3	7-12	10.82	16.18
WOD-MW004-2	16-GW4	6-11	10.81	14.94
WOD-MW006	16-GW6	4-9	11.05	9.66
WOD-MW008	16-GW8	18-28	30.61	13.35

Notes:
ft - feet
MSL – Mean Sea Level
BTOC – Below Top of Casing
Bg – below ground surface
Total depths based on July 2020 sampling event

TABLE 9-2
HISTORICAL GROUNDWATER ANALYTICAL RESULTS
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA
PAGE 1 OF 4

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW001 19970425	15-MW001 19980318	15-MW001 19980318 DUPLICATE	15-MW001 20030312	15-MW001 20081205	15-MW001 20081205	15-MW001 20100316	15-MW001 20100607	15-MW001 20100914	15-MW001 20101206	15-MW001 20110322	15-MW001 20110913	15-MW001 20120312	15-MW001 20120917	15-MW001 20130319
VOCs (ug/L)																
Benzene	5	10 U	2 J	10 U	1 U	1 U	1 U	0.3 U	0.3 U	0.3 J	0.8 J	0.3 U	1.08	0.5 U	0.2 U	0.11 U
Metals (ug/L)																
Total Arsenic	10	1.8 U	1.6 U	2	3 U	3.3	3.3	6.6	6.5 B	7.4 B	8.2	**10.9 J**	**13**	**11.7**	**12**	**11**
Dissolved Arsenic	NC	1.8 U	--		3 U	--	--	5.9	7.1 B	8.2 B	8.4	9.5 J	10.8	11.8	10	11
Field Parameters																
pH (S.U.)	NC	--	--	--	--	4.66	4.66	6.39	5.32	6.17	5.65	6.06	5.82	6.13	6.10	5.99
S. Conductivity (mS/cm)	NC	--	--	--	--	0.191	0.191	0.200	0.074	0.254	0.200	0.290	0.218	0.192	0.199	0.160
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	--	0.32	0.32	0.00	0.13	1.70	0.00	4.81	5.90	0.00	0.60	0.59
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	--	--	4.0	0.0	0.0	1.0
Temperature (°C)	NC	--	--	--	--	14.56	14.56	11.61	16.72	17.64	14.26	12.27	17.46	13.89	18.13	11.20
Oxygen Reduction Potential (mV)	NC	--	--	--	--	41	41	-45	-8	-55	-117	-57	-252.9	-16	2	-32
Turbidity (NTU)	NC	--	--	--	--	0.79	0.79	3.42	56.8	2.01	0.83	1.32	1.24	0.00	0.00	2.10

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW001 20130903	15-MW001 20140319	15-MW001 20140922	15-MW001 20150316	15-MW001 20150923	15-MW001 20160412	15-MW001 20160926	15-MW001 20171017	15-MW001 20200721
VOCs (ug/L)										
Benzene	5	0.25 U	0.25 U	--	--	--	--	--	--	--
Metals (ug/L)										
Total Arsenic	10	**13**	**11**	8 J	**12**	**15**	**18**	**20**	**21**	**30**
Dissolved Arsenic	NC	11	10	9.8	11	14	17	19	21	31
Field Parameters										
pH (S.U.)	NC	5.55	5.79	5.68	6.23	6.42	5.75	6.28	6.39	6.3
S. Conductivity (mS/cm)	NC	0.122	0.135	0.189	0.177	0.113	0.106	0.149	0.185	0.156
Dissolved Oxygen (mg/L) - Horiba	NC	0.00	0.00	0.68	2.46	0.90	0.00	0.00	0.00	0.00
Dissolved Oxygen (mg/L) - Test Kit	NC	0.1	0.4	0.0	0.2	1.0	0.05	0.00	0.00	0.00
Temperature (°C)	NC	20.58	10.86	17.03	10.76	18.54	15.3	17.44	16.38	16.56
Oxygen Reduction Potential (mV)	NC	175	4	-60	4	-15	30	-4	-17	-14
Turbidity (NTU)	NC	2.30	1.06	2.31	2.14	2.45	3.34	0.59	0.00	1.36

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW002 19970424	15-MW002 19980318	15-MW002 20030312	15-MW002 20081205	15-MW002 20081205	15-MW002 20100316	15-MW002 20100608	15-MW002 20100914	15-MW002 20101206	15-MW002 20110322	15-MW002 20110912	15-MW002 20120312	15-MW002 20120917	15-MW002 20130319	15-MW002 20130903
VOCs (ug/L)																
Benzene	5	--	--	1 U	1 U	1 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.5 U	0.5 U	0.2 U	0.11 U	0.25 U
Metals (ug/L)																
Total Arsenic	10	5	3.5	4.4 B	4.8 B	4.8 B	3.2 J	9.3 B	7.3 B	4.4 J	3.2 J	4.87 J	3 J	5	1.9	5.1 J
Dissolved Arsenic	NC	2.5	--	3.4 U	4.1 B	4.1 B	3.4 J	5.9 B	7.1 B	3.9 J	2.6 J	4.35 J	4 UL	6.6	1.7	4.8 J
Field Parameters																
pH (S.U.)	NC	--	--	--	5.49	5.49	4.65	6.1	5.34	5.16	5.63	5.23	5.59	5.3	4.53	5
S. Conductivity (mS/cm)	NC	--	--	--	0.000	0.000	0.086	0.199	0.114	0.074	0.101	0.107	0.134	0.118	0.125	0.093
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	9.01	9.01	0.00	0.00	2.98	0.00	5.51	1.88	0.00	0.63	0.12	0.00
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	--	1.0	0.0	1.0	0.2	0.1
Temperature (°C)	NC	--	--	--	12.61	12.61	9.71	15.64	19.55	13.48	10.89	19.86	11.95	23.07	10.80	20.41
Oxygen Reduction Potential (mV)	NC	--	--	--	52	52	-56	-31	14	-73	-13	68.3	43	9	64	260
Turbidity (NTU)	NC	--	--	--	16.00	16.00	36.8	1.32	3.01	17.6	21.1	8.59	27.2	9.40	81.7	3.72

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW002 20140319	15-MW002 20140922	15-MW002 20150316	15-MW002 20150923	15-MW002 20160412	15-MW002 20160926	15-MW002 20171017	15-MW002 20200721
VOCs (ug/L)									
Benzene	5	0.25 U	--	--	--	--	--	--	--
Metals (ug/L)									
Total Arsenic	10	2.8 J	3.2 J	1.6	4.3 J	5 U	4.8 J	5.5	5.4
Dissolved Arsenic	NC	2.5 U	4	3.1	4 J	2.9 U	7	4.4 J	5.4
Field Parameters									
pH (standard units)	NC	5.55	5.78	5.85	5.95	5.63	5.83	6.21	6.33
S. Conductivity (millisiemens per centimeter)	NC	0.085	0.107	0.122	0.067	0.119	0.059	0.15	0.092
Dissolved Oxygen (mg/L) - Horiba	NC	0.00	2.16	3.20	0.94	0.00	0.00	0.00	19.26
Dissolved Oxygen (mg/L) - Test Kit	NC	0.6	2.0	1.0	1.0	1.0	1.0	0.1	9.0
Temperature (degrees Celsius)	NC	8.31	18.39	8.86	19.66	13.51	20.77	17.67	19.55
Oxygen Reduction Potential (millivolts)	NC	19	-7	11	58	-9	44	-11	6
Turbidity (nephelometric turbidity units)	NC	52.9	14.80	100.5	9.03	7.31	9.02	0.00	5.12

Notes:

NC - no criteria

-- - not analyzed

µg/L - micrograms per liter water

J - Estimated Value

L - Biased Low

U - Analyte was not detected in the sample at a level greater than the instument detection

R - Surrogate Recovery Noncompliance

mg/L - milligrams per liter

Bolded and shaded cells indicate exceedances of the Cleanup Goal

TABLE 9-2
HISTORICAL GROUNDWATER ANALYTICAL RESULTS
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA
PAGE 2 OF 4

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW007 19930314	15-MW007 19970425	15-MW007 19980318	15-MW007 20000218	15-MW007 20030314	15-MW007 20081208	15-MW007 20090114	15-MW007 20100316	15-MW007 20100608	15-MW007 20100608 duplicate
VOCs (ug/L)											
Benzene	5	--	**42**	**23 K**	**58**	**11**	**32**	2	0.3 U	0.3 U	0.3 J
Metals (ug/L)											
Total Arsenic	10	**15.6**	**68.8**	**88.2**	**60.4**	**15.6 B**	**27**	9.7	3.9 J	**15.5 J**	**27.7 J**
Dissolved Arsenic	NC	15.1				15.1 B	--	--	4.6 J	22.4 J	22.4 J
Field Parameters											
pH (S.U.)	NC	--	--	--	--	--	5.37	4.18	5.54	6.06	--
S. Conductivity (mS/cm)	NC	--	--	--	--	--	0.173	0.100	0.087	0.111	--
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	--	--	0.73	4.12	0.00	2.35	--
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	--
Temperature (°C)	NC	--	--	--	--	--	16.38	15.70	11.90	17.36	--
Oxygen Reduction Potential (mV)	NC	--	--	--	--	--	-192	58	-35	-132	--
Turbidity (NTU)	NC	--	--	--	--	--	9.15	4.46	7.50	5.76	--

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW007 20100915	15-MW007 20101206	15-MW007 20101206 duplicate	15-MW007 20110321	15-MW007 20110321 duplicate	15-MW007 20110912	15-MW007 20110912 duplicate	15-MW007 20120312	15-MW007 20120312 duplicate	15-MW007 20120917
VOCs (ug/L)											
Benzene	5	**33**	2	1	4	5	**11.1**	**14.2**	**12**	**11**	2.4 J
Metals (ug/L)											
Total Arsenic	10	**54.4**	**24.5**	**23.8**	**58 J**	**39.9 J**	**46.6**	**45.9**	**46.8**	**57.4**	**19**
Dissolved Arsenic	NC	39.7	28.5	26.8	48.5 J	34.6 J	45.6	47.1	**60.5**	**60.4**	18
Field Parameters											
pH (S.U.)	NC	6.08	5.31	--	6.37	--	5.42	--	6.00	--	6.02
S. Conductivity (mS/cm)	NC	0.446	0.142	--	0.137	--	0.212	--	0.288	--	0.163
Dissolved Oxygen (mg/L) - Horiba	NC	1.62	0.59	--	--	--	2.24	--	0.00	--	0.72
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	0.0	--	1.0	--	1.0
Temperature (°C)	NC	18.60	15.40	--	16.60	--	19.08	--	16.85	--	20.24
Oxygen Reduction Potential (mV)	NC	-118	-132	--	-87	--	-237.6	--	-110	--	-7.6
Turbidity (NTU)	NC	3.19	2.75	--	1.71	--	2.20	--	1.87	--	3.85

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW007 20120917 duplicate	15-MW007 20130318	15-MW007 20130318 duplicate	15-MW007 20130903	15-MW007 20130903 duplicate	15-MW007 20140319	15-MW007 20140319 duplicate	15-MW007 20140922	15-MW007 20140922 duplicate	15-MW007 20150316
VOCs (ug/L)											
Benzene	5	2.1 J	0.11 U	0.11 U	0.51 J	0.54 J	0.5 J	0.46 J	--	--	--
Metals (ug/L)											
Total Arsenic	10	**16**	0.29 U	0.51 J	3.3 J	2.8 J	4.1 J	4 J	3.3 J	4.3 J	10
Dissolved Arsenic	NC	22	1.9	0.29 U	3.2 J	2.9 J	3.3 U	3.4 U	1.7	2.9	9.1
Field Parameters											
pH (S.U.)	NC	--	4.61	--	4.95	--	5.12	--	5.15	--	5.55
S. Conductivity (mS/cm)	NC	--	0.096	--	0.074	--	0.094	--	0.138	--	0.168
Dissolved Oxygen (mg/L) - Horiba	NC	--	0.55	--	5.09	--	0.00	--	0.97	--	1.82
Dissolved Oxygen (mg/L) - Test Kit	NC	--	0.4	--	4.0	--	0.3	--	0.8	--	2.0
Temperature (°C)	NC	--	12.67	--	21.71	--	15.12	--	17.90	--	15.24
Oxygen Reduction Potential (mV)	NC	--	101	--	-122	--	-5	--	6	--	-42
Turbidity (NTU)	NC	--	23.8	--	3.02	--	4.31	--	1.49	--	7.44

LOCATION **SAMPLE DATE**	**Cleanup Goal**	15-MW007 20150316 duplicate	15-MW007 20150923	15-MW007 20150923 duplicate	15-MW007 20160412	15-MW007 20160412 duplicate	15-MW007 20160926	15-MW007 20160926 duplicate	15-MW007 20160926	15-MW007 20160926 duplicate	15-MW007 20200720	
VOCs (ug/L)												
Benzene	5	--	--	--	--	--	--	--	--	--	--	
Metals (ug/L)												Duplicate
Total Arsenic	10	10	5.4	6.1	3.4 U	6.4	3.4 J	3 J	6.4	5.6	**51**	**59**
Dissolved Arsenic	NC	9.1	5 J	5.4	3.9 U	4.3 U	3.6 J	6.2	4.4 J	4.9 J	54	56
Field Parameters												
pH (standard units)	NC	--	5.63	--	4.67	--	5.17	--	5.46	--	6.34	--
S. Conductivity (millisiemens per centimeter)	NC	--	0.097	--	0.073	--	0.081	--	0.125	--	0.266	--
Dissolved Oxygen (mg/L) - Horiba	NC	--	0.00	--	0.00	--	0.00	--	0.00	--	0.00	--
Dissolved Oxygen (mg/L) - Test Kit	NC	--	0.3	--	0.0	--	0.0	--	0.1	--	0.0	--
Temperature (degrees Celsius)	NC	--	20.95	--	15.79	--	21.51	--	17.49	--	21.67	--
Oxygen Reduction Potential (millivolts)	NC	--	73	--	89	--	-37	--	35	--	-79	--
Turbidity (nephelometric turbidity units)	NC	--	1.53	--	13.4	--	5.49	--	0.00	--	2.04	--

Notes:

NC - no criteria

-- - not analyzed

µg/L - micrograms per liter water

J - Estimated Value

L - Biased Low

U - Analyte was not detected in the sample at a level greater than the instument detection

R - Surrogate Recovery Noncompliance

mg/L - milligrams per liter

Bolded and shaded cells indicate exceedances of the Cleanup Goal

TABLE 9-2
HISTORICAL GROUNDWATER ANALYTICAL RESULTS
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA
PAGE 3 OF 4

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW002S 19980528	WOD-MW002S 20000218	WOD-MW002S 20030312	WOD-MW002S 20081205	WOD-MW002S 20100316	WOD-MW002S 20100607	WOD-MW002S 20100915	WOD-MW002S 20101207	WOD-MW002S 20110321	WOD-MW002S 20110913	WOD-MW002S 20120312	WOD-MW002S 20120917	WOD-MW002S 20130318	WOD-MW002S 20130903	WOD-MW002S 20140319
VOCs (ug/L)																
Benzene	5	10 U	10 U	1 U	5	0.3 U	0.4 J	1	1 J	0.3 U	0.5 U	0.5 U	0.17 J	0.11 U	0.25 U	0.25 U
Metals (ug/L)																
Total Arsenic	10	**11**	**18.2**	**12.5 B**	**18.4**	4.2 J	**15.3 B**	**26.9**	**18.2**	4 J	5.72	4 UL	5.3	0.29 U	5.4 J	1.7 J
Dissolved Arsenic	NC	--	--	9.5 B	16	1.5 U	19.2 J	23.1	16	6.7 J	3 U	4 UL	11	2	4.4 J	1.3 U
Field Parameters																
pH (S.U.)	NC	--	--	--	6.44	6.43	5.77	6.65	6.40	6.02	3.12	6.56	6.45	6.73	6.35	6.45
S. Conductivity (mS/cm)	NC	--	--	--	0.233	0.195	0.380	0.320	0.225	0.240	0.268	0.278	0.242	0.313	0.141	0.244
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	5.73	0.00	10.02	3.73	0.00	--	1.91	1.45	2.86	3.15	1.91	2.44
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	4.0	1.0	1.5	1.0	2.0	3.0
Temperature (°C)	NC	--	--	--	13.95	11.57	17.01	20.65	13.14	14.50	20.06	15.89	21.06	10.52	19.20	11.62
Oxygen Reduction Potential (mV)	NC	--	--	--	-14	-15	-72	-101	-88	-15	697	9	-4	9	-26	28
Turbidity (NTU)	NC	--	--	--	19.0	10.50	24.1	1.35	78.2	32.5	41.1	37.1	6.39	4.03	19.2	17.76

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW002S 20140922	WOD-MW002S 20150316	WOD-MW002S 20150923	WOD-MW002S 20160412	WOD-MW002S 20160926	WOD-MW002S 20171017
VOCs (ug/L)							
Benzene	5	--	--	--	--	--	--
Metals (ug/L)							
Total Arsenic	10	2.8 J	3.8	7.3	3.8 U	2.9 J	2.3 U
Dissolved Arsenic	NC	6.6	2.3	5.5	2.3 U	2.3 U	2.3 U
Field Parameters							
pH (S.U.)	NC	6.06	6.76	6.85	6.55	6.61	6.81
S. Conductivity (mS/cm)	NC	0.151	0.286	0.155	0.217	0.224	0.247
Dissolved Oxygen (mg/L) - Horiba	NC	1.67	3.13	0.44	2.00	0.00	1.78
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	1.0	2.0	--	1.5
Temperature (°C)	NC	17.62	13.72	20.17	20.46	21.7	17.1
Oxygen Reduction Potential (mV)	NC	-27	-12	57	33	-3	-9
Turbidity (NTU)	NC	11.70	47.2	9.61	9.87	--	0.00

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW002D 19980528	WOD-MW002D 19980528 DUPLICATE	WOD-MW002D 20000218	WOD-MW002D 20030312	WOD-MW002D 20081205	WOD-MW002D 20100316	WOD-MW002D 20100316 duplicate	WOD-MW002D 20100607	WOD-MW002D 20100914	WOD-MW002D 20101207	WOD-MW002D 20110321	WOD-MW002D 20110913	WOD-MW002D 20120312	WOD-MW002D 20120917	WOD-MW002D 20130318
VOCs (ug/L)																
Benzene	5	**22**	**23**	**25**	**8**	3	**8**	**8**	1 J	0.6 J	0.3 U	3	2.57	2.2	1.9	4.5
Metals (ug/L)																
Total Arsenic	10	**27.5**	**29.2**	**30.2**	**21.4**	**12.3**	**10.9**	**10.5**	8.4 B	**10.6**	9.6	8.7 J	**11.2**	**11**	**14**	**13**
Dissolved Arsenic	NC	--	--	--	19.5	--	10.5	11.4	6.8 B	10	9.1	7.6 J	9.03	9.6 L	9.8	9
Field Parameters																
pH (S.U.)	NC	--	--	--	--	6.17	7	--	5.71	7.22	6.19	6.02	4.66	6.22	6.17	6.25
S. Conductivity (mS/cm)	NC	--	--	--	--	0.168	0.327	--	62.500	0.126	0.118	0.117	0.154	0.167	0.186	0.237
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	--	1.84	3.96	--	4.40	15.58	2.63	--	1.17	0.00	0.58	0.01
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	--	--	0.15	0.0	0.5	0.1
Temperature (°C)	NC	--	--	--	--	15.16	11.15	--	17.15	18.40	16.10	15.20	18.12	17.22	19.00	11.66
Oxygen Reduction Potential (mV)	NC	--	--	--	--	-19	17	--	-72	-65	16	-44	113.2	-39	-10	-34
Turbidity (NTU)	NC	--	--	--	--	0.70	4.09	--	0.71	0.62	0.54	1.32	0.41	0.21	8.04	17.30

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW002D 20130903	WOD-MW002D 20140319	WOD-MW002D 20140922	WOD-MW002D 20150316	WOD-MW002D 20150923	WOD-MW002D 20160411	WOD-MW002D 20160926	WOD-MW002D 20171017	WOD-MW002D 20200720
VOCs (ug/L)										
Benzene	5	2 J	1.2 J	--	--	--	--	--	--	--
Metals (ug/L)										
Total Arsenic	10	**16**	**11**	9 J	9.1	**11**	**16**	**14**	**11**	6.2
Dissolved Arsenic	NC	15	8.8 J	8.7	9.5	11	15	14	9.4	7.2
Field Parameters										
pH (standard units)	NC	6.05	6.23	5.78	6.23	6.42	6.33	6.36	6.25	5.86
S. Conductivity (millisiemens per centimeter)	NC	0.115	0.159	0.176	0.145	0.105	0.137	0.125	0.134	0.11
Dissolved Oxygen (mg/L) - Horiba	NC	0.20	0.00	1.09	1.19	0.00	0.00	0.00	0.00	0.80
Dissolved Oxygen (mg/L) - Test Kit	NC	< 1	0.6	3.0	0.3	0.3	0	0.1	0.1	4.0
Temperature (degrees Celsius)	NC	21.68	13.05	17.43	14.17	17.29	15.81	19.98	15.07	17.92
Oxygen Reduction Potential (millivolts)	NC	-83	-33	-10	-7	22	-12	-38	-7	36
Turbidity (nephelometric turbidity units)	NC	7.58	24.6	1.11	5.83	0.53	2.14	0.14	0.00	2.90

Notes:

NC - no criteria
-- - not analyzed
µg/L - micrograms per liter water
J - Estimated Value
L - Biased Low
U - Analyte was not detected in the sample at a level greater than the instument detection
R - Surrogate Recovery Noncompliance
mg/L - milligrams per liter
Bolded and shaded cells indicate exceedances of the Cleanup Goal

TABLE 9-2
HISTORICAL GROUNDWATER ANALYTICAL RESULTS
WASTE OIL DUMP
NASA WALLOPS FLIGHT FACILITY
WALLOPS ISLAND, VIRGINIA
PAGE 4 OF 4

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW003A 19970425	WOD-MW003A 19980319	WOD-MW003R 20030312	WOD-MW003R 20090114	WOD-MW003R 20100315	WOD-MW003R 20100608	WOD-MW003R 20100915	WOD-MW003R 20101207	WOD-MW003R 20110322	WOD-MW003R 20110913	WOD-MW003R 20120312	WOD-MW003R 20120917	WOD-MW003R 20130318	WOD-MW003R 20130903	WOD-MW003R 20140319
VOCs (ug/L)																
Benzene	5	--	--	1 U	1 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.5 U	0.5 U	0.2 U	0.11 U	0.25 U	0.25 U
Metals (ug/L)																
Total Arsenic	10	5.1	1.6 U	3 U	1.45	1.5 U	2.2 B	1.6 B	0.8 U	2.25 UJ	1.5 U	4 UL	0.5 U	1.2	1.2 U	1.2 U
Dissolved Arsenic	NC	--	--	3 U	--	1.5 U	3.1 B	0.8 U	0.8 U	2.25 UJ	1.5 U	4 UL	0.5 U	0.75 J	1.2 U	1.2 U
Field Parameters																
pH (S.U.)	NC	--	--	--	5.65	6.69	6.38	5.14	5.91	6.13	4.40	6.34	5.98	6.26	6.01	5.80
S. Conductivity (mS/cm)	NC	--	--	--	0.170	0.132	0.100	0.131	0.127	0.090	0.107	0.116	0.107	0.119	0.056	0.061
Dissolved Oxygen (mg/L) - Horiba	NC	--	--	--	8.56	11.9	10.02	19.99	11.01	--	9.18	6.52	5.42	7.74	5.61	5.99
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	--	--	--	9.0	6.0	5.0	10.0	6.0	6.0
Temperature (°C)	NC	--	--	--	15.30	13.44	16.09	19.40	14.66	15.40	18.36	18.60	19.21	13.09	22.49	14.60
Oxygen Reduction Potential (mV)	NC	--	--	--	75	239	370	49	156	159	615.5	147	158	354	173	208
Turbidity (NTU)	NC	--	--	--	1.18	0.33	5.30	5.99	2.42	4.28	7.42	6.53	7.32	0.60	7.14	0.17

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW003R 20140922	WOD-MW003R 20150316	WOD-MW003R 20150923	WOD-MW003R 20160412	WOD-MW003R 20160926	WOD-MW003R 20171017	WOD-MW003R 20200721
VOCs (ug/L)								
Benzene	5	--	--	--	--	--	--	--
Metals (ug/L)								
Total Arsenic	10	0.29 UJ	0.29 U	2.3 U	2.3 U	2.3 U	2.3 U	3 U
Dissolved Arsenic	NC	0.29 U	0.34 J	2.3 U	4.2 U	2.3 U	2.3 U	3 U
Field Parameters								
pH (S.U.)	NC	6.02	6.15	6.25	5.71	5.88	6.38	5.95
S. Conductivity (mS/cm)	NC	0.067	0.073	0.044	0.075	0.049	0.106	0.089
Dissolved Oxygen (mg/L) - Horiba	NC	5.85	7.23	8.00	3.00	4.64	6.99	8.27
Dissolved Oxygen (mg/L) - Test Kit	NC	5.0	3.0	8.0	3.0	3.0	6.0	7.0
Temperature (°C)	NC	18.20	13.82	18.20	15.56	21.76	17.28	19.22
Oxygen Reduction Potential (mV)	NC	200	240	184	159	188	145	258
Turbidity (NTU)	NC	5.26	0.93	1.80	1.44	4.30	0.00	0.00

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW008 20081210	WOD-MW008 20100315	WOD-MW008 20100607	WOD-MW008 20100914	WOD-MW008 20101206	WOD-MW008 20110321	WOD-MW008 20110913	WOD-MW008 20120312	WOD-MW008 20120917	WOD-MW008 20130318	WOD-MW008 20130903	WOD-MW008 20140319	WOD-MW008 20140922	WOD-MW008 20150316	WOD-MW008 20150923
VOCs (ug/L)																
Benzene	5	1 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.5 U	0.5 U	0.2 U	0.11 U	0.25 U	0.25 U	--	--	--
Metals (ug/L)																
Total Arsenic	10	6.3	1.5 U	4.9 B	1 B	0.8 U	2.25 UJ	1.5 U	4 UL	0.5 U	0.29 U	1.2 U	1.2 U	0.29 UJ	0.34 J	2.3 U
Dissolved Arsenic	NC	--	1.5 U	4.7 B	2.7 B	0.8 U	2.25 UJ	1.5 U	4 UL	0.5 U	0.29 U	1.2 U	1.2 U	1.7	0.29 U	2.3 U
Field Parameters																
pH (S.U.)	NC	5.77	6.29	5.7	4.35	6.09	5.61	3.26	6	5.74	5.34	4.91	5.44	5.27	5.84	6.06
S. Conductivity (mS/cm)	NC	0.120	0.098	0.090	0.094	0.104	0.096	0.081	0.085	0.102	0.097	0.060	0.098	0.102	0.109	0.069
Dissolved Oxygen (mg/L) - Horiba	NC	1.98	3.75	6.23	18.82	9.89	10.67	13.78	11.94	6.66	7.69	6.65	5.43	6.04	6.10	7.47
Dissolved Oxygen (mg/L) - Test Kit	NC	--	--	--	--	--	--	> 12	10.0	6.0	2.0	7.0	5.0	6.0	3.0	5.0
Temperature (°C)	NC	15.17	11.87	15.01	18.80	16.71	15.04	18.00	14.78	18.48	10.98	22.71	13.22	15.70	12.93	17.56
Oxygen Reduction Potential (mV)	NC	84	132	358	138	294	262	-170.2	267	253	231	366	222	205	172	234
Turbidity (NTU)	NC	7.10	1.56	2.01	0.79	1.20	0.00	0.62	0.00	8.31	0.15	1.70	2.98	0.48	0.26	1.54

LOCATION **SAMPLE DATE**	**Cleanup Goal**	WOD-MW008 20160411	WOD-MW008 20160926	WOD-MW008 20171017	WOD-MW008 20200720
VOCs (ug/L)					
Benzene	5	--	--	--	--
Metals (ug/L)					
Total Arsenic	10	2.3 U	2.3 U	2.3 U	3 U
Dissolved Arsenic	NC	2.3 U	2.3 U	2.3 U	3 U
Field Parameters					
pH (standard units)	NC	6.35	5.94	6.11	6.22
S. Conductivity (millisiemens per centimeter)	NC	0.080	0.091	0.1	0.087
Dissolved Oxygen (mg/L) - Horiba	NC	2.40	0.90	6.61	8.62
Dissolved Oxygen (mg/L) - Test Kit	NC	1.0	2.0	6.0	6.0
Temperature (degrees Celsius)	NC	15.10	17.51	15.82	18.64
Oxygen Reduction Potential (millivolts)	NC	83	305	216	319
Turbidity (nephelometric turbidity units)	NC	0.29	0.33	0.00	0.00

Notes:
NC - no criteria
-- - not analyzed
µg/L - micrograms per liter water
J - Estimated Value
L - Biased Low
U - Analyte was not detected in the sample at a level greater than the instument detection
R - Surrogate Recovery Noncompliance
mg/L - milligrams per liter
Bolded and shaded cells indicate exceedances of the Cleanup Goal

SAP Worksheet #10: Problem Statement

(UFP-QAPP Manual Section 2.6.2 - Worksheet #10)

10.1 PROBLEM STATEMENT

Based on human health and ecological risk assessments and exceedances of regulatory standards, COCs were identified at the Waste Oil Dump (WOD) Site at the NASA Goddard Space Flight Center WFF located in Accomack County, Virginia. The sole medium of concern at the WOD is shallow groundwater in the upper, unconfined zone of the Columbia aquifer; past disposal operations at the WOD are the likely source of groundwater contamination.

This LTM Plan – Revision 4 details the procedures to be used during the sampling and analysis of groundwater collected as a component of the remedial action being implemented. The objective of the monitoring program for the WOD is to document the concentrations in site groundwater contaminants and monitor progress in attaining the cleanup goals identified in the ROD signed in March 2008. Currently, arsenic is the only analytical parameter included in the groundwater LTM program for the WOD.

10.2 STUDY GOALS

The LTM goals are as follows:

- Collect and analyze groundwater samples from three existing wells to monitor progress towards attaining the cleanup goal for the remaining COC, arsenic.
- Collect water level measurements and field parameters from twelve wells to confirm groundwater flow at the Site.

10.3 INFORMATION INPUTS

The principal inputs to meet the study goals above are analytical chemistry data obtained from groundwater, as well as field groundwater geochemical analyses.

Data required to fulfill these goals are:

- Assessment of three monitoring wells for concentrations of total and dissolved arsenic. The EPA National Primary Drinking Water Regulations MCL for arsenic (10 micrograms per liter [µg/L]) will be used as the project action limit (PAL) for groundwater.
- Field measurements (conductivity, temperature, pH, turbidity, dissolved oxygen [DO], and oxidative-reductive potential [ORP]) at three wells to determine the ambient physical and chemical properties of the groundwater at the time of sampling.
- Water level measurements will be collected from twelve wells during each sampling event and will be used to confirm groundwater flow.
- Groundwater sampling for analytical chemistry and field geochemistry will occur twice every five years or until project objectives are met.

10.4 STUDY BOUNDARIES

Groundwater samples for total/dissolved arsenic analysis and water level measurements and field geochemical parameters (dissolved oxygen, temperature, pH, conductivity, turbidity, and ORP) will be collected at the following three existing monitoring wells: 15-MW001, 15-MW007, and WOD-MW002D. Water level measurements will be collected from the following twelve wells: 15-MW001, 15-MW002, 15-

MW003, 15-MW007, WOD-MW001, WOD-MW002S, WOD-MW002D, WOD-MW003, WOD-MW003R, WOD-MW004-2, WOD-MW006, and WOD-MW008.

10.5 ANALYTICAL APPROACH

Worksheet #13 presents the sampling plan details. The Limit of Quantitation (LOQ) to be met is provided in Worksheet #16. Groundwater samples will be analyzed for total and dissolved arsenic by EPA SW-846 6020A. The sample volume collected for the dissolved metals analysis will be field-filtered on-site.

10.6 PERFORMANCE/ACCEPTANCE CRITERIA

Defined project specific measurement performance criteria have been established for precision, accuracy, sensitivity, and completeness. Requirements for precision, accuracy, representativeness and comparability are presented in Worksheets #11 and #18. Requirements for analytical sensitivity are presented in Worksheet #16. Project completeness goals have been set at 95% for both field and laboratory completeness as defined in Worksheet #19, Data Usability Assessment.

Procedures for data validation are outlined in Worksheet #19. All groundwater data will be validated to achieve the Stage 4 level of validation. Data validation tasks will include evaluation of field and laboratory achieved performance for each project specific measurement performance criterion. The findings of these evaluations will be included in the corresponding data validation reports. After validation, project team members will evaluate data usability and will assess whether the data can be used as intended to support the attainment of project objectives.

10.7 SAMPLING DESIGN/PLAN FOR OBTAINING DATA

Long Term Monitoring will consist of the collection and analysis of groundwater samples from monitoring wells within the contaminant plume to assess the progress towards attaining the cleanup goal. Groundwater samples will be collected twice every five years for laboratory analysis and field parameters as outlined in section 10.4. The wells will be purged with a peristaltic pump using low flow purging techniques. The field data will be recorded in field data collection sheets and field logbooks. Discrete grab groundwater samples will be collected using a peristaltic pump and will be shipped to Eurofins TestAmerica Pittsburgh for total and dissolved metals analysis. The laboratory will provide analytical data in the form of a Level IV data package.

Monitoring will continue until four consecutive monitoring events confirm that the clean-up level has been attained in the designated monitoring wells that are included in this SAP.

SAP Worksheet #11: Field Quality Control Samples

(UFP-QAPP Manual Section 2.6.2-Worksheet #12)

Quality Control (QC) Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)
Field Duplicate (FD)	Total Metals, Dissolved Metals	1 per sampling event (1 per 10 samples)	Precision	Relative Percent Difference (RPD) ≤ 30% when detected in both samples at a concentration ≥ 5X LOQ. If the analyte concentration in either the parent sample or field duplicate sample is < 5X LOQ, then the absolute difference of the results must be ≤ LOQ.
Field Blank	Total Metals, Dissolved Metals	1 per sampling event	Accuracy/Bias/Contamination	No target analyte concentrations ≥ 1/2 LOQ.
Cooler Temperature Blank	N/A	1 per cooler	Representativeness	Temperature must be between 0 °C and 6 °C

SAP Worksheet #12: Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1-Worksheet #14)

12.1 Long Term Monitoring Task Plan

The following is a list of field tasks that will be performed for the investigation. A short description of each task is provided under Section 12.2.

- Mobilization/Demobilization and Safety Training
- Equipment Calibration
- Groundwater Sampling
- Water Level Measurements
- Survey Locations
- Decontamination
- Investigation Derived Waste (IDW) Management
- Documentation

The following is a list of additional project-related analytical and reporting tasks that will be completed as part of this investigation. A short description of each task is provided under **Section 12.3**.

- Analytical Tasks
- Data Handling and Management
- Data Tracking
- Data Storage, Archiving, and Retrieval
- Data Security
- Electronic Data
- Data Review and Validation
- Project Reports

Bluestone Standard Operating Procedures (SOPs) and field forms for field tasks referenced in this section are identified by title in Worksheet #14, and copies of applicable SOPs are provided in Appendix A. Field activities will be conducted in accordance with Bluestone SOPs. Field sampling equipment, including oil/water interface meters, water level indicators, non-disposable bailers (brought into the field as a backup sample collection method), and other nondedicated equipment used at each sample location will be cleaned between uses as provided in Bluestone SOP #05.

12.2 Field Tasks

A summary of each field task is provided in the following sections.

12.2.1 Mobilization/Demobilization and Safety Training

Mobilization will consist of the delivery of equipment, materials, and supplies to the site, the complete assembly in satisfactory working order of such equipment at the site, and the satisfactory storage at the site of such materials and supplies.

Bluestone will prepare a list of equipment and supplies necessary for the field team to perform the field activities. The necessary equipment and supplies include, but are not limited to, the following:

- Documents, forms, logbooks, log sheets, labels, custody seals, air bills, and other paperwork required by the SAP and contract wide approved Health and Safety Plan.
- Vehicles for personnel, equipment, and sample transport.

- Personnel, supplies, and equipment (e.g., bottle ware and personal protective equipment [PPE]) required by the SAP and project-specific HASP.
- Required sample containers.
- Equipment and supplies for sample custody, preservation, and packaging.
- Other miscellaneous office and field supplies.

Project-specific health and safety training for all Bluestone field staff and subcontractors, as applicable, will be provided as part of site mobilization and is also addressed in Worksheet #7. During the required training and orientation, field team members will review the SAP and will be given any project-specific health and safety training based on the HASP. Field personnel must review and sign off on the project specific HASP. The SSHO will be responsible for reviewing the HASP with field team members. It will also be necessary to provide orientation and health and safety training for any additional or replacement field team members assigned after mobilization.

The Bluestone field representative will also coordinate with the Facility POC, Ms. Susan Dunn, verbally or via email at least 1 week prior to commencement of field work to arrange for access to the site and to identify appropriate locations for the temporary storage of equipment and supplies.

Demobilization will consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the investigation. Upon demobilization, field investigation paperwork will be filed and docketed in the project file.

12.2.3 Equipment Calibration

Field equipment will be calibrated at the beginning of each day, unless otherwise stated by the equipment manufacturer or if problems are noted in the field. These procedures are described in Worksheet #15.

12.2.5.3 *Groundwater Sampling*

Groundwater samples will be collected from three wells. Groundwater samples will be collected for arsenic. A sample log sheet will be created to document the details of each sample. Groundwater sampling activities will be conducted in accordance with Bluestone SOP #12 (Appendix A).

Water quality parameters will be monitored and recorded on field log forms during sampling activities for groundwater samples and the groundwater grab samples. Sampling may be conducted once three consecutive readings, taken at 5 to 10-minute intervals, are within the following limits:

- pH ±0.2 standard units
- Specific conductance ±10%
- Temperature ±10%
- Turbidity less than 10 NTUs
- DO ±10%
- ORP ±10%
- A flow rate between 100 and 500 ml/min

12.2.6 Water Level Measurements

Groundwater level measurements will be collected following the procedures outlined in Bluestone SOPs (Appendix A). Water level measurements will be completed within the shortest time possible on the same day, and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or north side of the well casing. The measurement will be decontaminated prior to conducting the measurement and between each well.

12.2.7 Survey Locations

Survey locations will not be collected at this time.

12.2.8 Decontamination

Decontamination will be required for all reusable sampling/measuring equipment to prevent transferring potential contaminants to other locations or samples. Reusable sampling/measuring equipment will be decontaminated with a non-phosphate detergent scrub followed by a per- and polyfluoroalkyl substances -free water rinse. The water used to decontaminate the tooling and equipment will be NASA potable water and all fluids generated during decontamination will be contained and managed as IDW.

Decontamination procedures are detailed in Bluestone SOP #05 (Appendix A).

12.2.9 Investigation Derived Waste Management

IDW will consist of decontamination water and purge water. IDW will be containerized in 55-gallon drums or bulk storage container, properly labeled, sampled, and characterized. The temporary IDW storage location is Building B-29 for the Main Base. The disposal of aqueous IDW will be coordinated by Bluestone.

If contaminants of concern concentration levels in IDW are over the PSL list, the aqueous IDW will be prepared for disposal off-site.

Used PPE (i.e., nitrile gloves), soil sleeves, and general refuse will be bagged and disposed as general trash.

12.2.10 Documentation

Sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded in bound field notebooks (waterproof logbooks will not be used) or on specific field forms. Samples will be packaged and shipped according to Bluestone SOP #10 and SOP #13 in Appendix A.

A summary of field activities will be properly recorded in a bound logbook with consecutively numbered pages. Logbooks will be assigned to field personnel and stored in a secured area when not in use. Logbooks will be maintained in accordance with Bluestone SOP #06 (Appendix A).

Entries will be written in ink. No erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change. Sampling forms, and other field forms will also be used to document field activities.

12.3 ANALYTICAL AND REPORTING TASKS

The following is a list of analytical and data management/reporting tasks.

12.3.1 Analytical Tasks

Chemical analyses of Analytes in all media will be performed by Eurofins TestAmerica located in Pittsburgh, PA. Eurofins Pittsburgh has been accredited to conduct the analyses required by this SAP by the of Virginia Environmental Laboratory Accreditation Program (VELAP). A copy of Eurofins Pittsburgh's accreditation is provided in Appendix C. Analyses for Arsenic will be performed in accordance with the analytical methods specified in Worksheet #13. The laboratory will perform chemical analysis following laboratory-specific SOPs (Worksheet #17) based on the analytical methods listed in Worksheet #13. Laboratory data will be delivered to Bluestone in the form of an electronic data deliverable (EDD) and portable document format (PDF) data package. This information will be captured in the project database that will be uploaded upon completion of the report.

12.3.2 Data Handling and Management

After the sampling event is complete, the field sampling log sheets will be organized by date and medium and filed in the project files. The field logbooks for this project will be used only for this facility and will also be categorized and maintained in the project files after completion of the field program. The field logbooks will be titled based on date and activity.

Prior to placement in the project files, the logbook pages will be scanned electronically. The data handling procedures to be followed by the laboratory will meet the requirements of the laboratory technical specification. The electronic data will be automatically downloaded accordance with Bluestone SOPs.

12.3.3 Data Tracking

Data will be tracked from generation to archiving in the project-specific files. The Bluestone QAM/Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted analytical laboratory. Upon receipt of the data packages from the analytical laboratory, the project chemist will coordinate the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

12.3.4 Data Storage, Archiving, and Retrieval

After the data are validated, the data packages are entered into the Bluestone and NASA file systems and archived in secure files. The field records including field logbooks, sample log sheets, and chain-of-custody records will be submitted by the Bluestone field representative to be entered into the Bluestone and NASA file systems prior to archiving in secure project files. As documents are finalized, all relevant data and records are uploaded electronically to the project database and retained there indefinitely.

12.3.5 Data Security

The Bluestone project files are restricted to designated personnel only. The Bluestone Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

12.3.6 Electronic Data

One hundred percent (100%) of the laboratory data (electronic and PDF report) will be validated in accordance with specifications in Worksheets #19, and qualifiers will be manually added to the database. Data will then be compiled and loaded into the project database. This process includes a QA review of the data to ensure that the content and format of the data satisfy Bluestone requirements.

12.3.7 Data Review and Validation

Data verification, validation, and usability assessment processes are described on Worksheets #19.

12.3.8 Project Reporting

After completion of the data review project-specific reports will be prepared to present results of the investigations along with conclusions and recommendations. The draft report will be submitted to the NASA RPM for initial review, and NASA comments will be addressed.

SAP Worksheet #13: Sample Details Table

(UFP-QAPP Manual Section 2.8.1-Worksheets #18,19,20 and 30)

Waste Oil Dump **Wallops Flight Facility,** **Wallops Island, Virginia** **(Tentative Sampling Dates – September 2022 and twice per 5 years)** **Eurofins TestAmerica Pittsburgh** **Sample Receiving** **301 Alpha Drive** **Pittsburgh, PA 15238** Project Manager: Andy Johnson Phone: 615-818-9567 Email: Andy.Johnson@Eurofinset.com	**Analysis Group**	Arsenic (total and dissolved)
	Preparation and Analytical Method	Arsenic: SW-846 3005A/SW-846 6020A
	Analytical Laboratory/ Analytical SOP Reference	Eurofins SOP: PT-IP-003/PT-MT-002
	Data Package Turnaround Time	21 Calendar Days
	Container Type/ Volume Required (if different than container volume)	Arsenic: 1-250 milliliter (mL) polyethylene bottle
	Preservative	Arsenic: Nitric acid to pH<2 (Dissolved samples must be field-filtered through a 0.45-micron pore diameter membrane prior to preservation)
	Holding Time (Preparation/Analysis)	Arsenic: 6 months to analysis

Proposed Groundwater Samples				
Site	**Matrix**	**Sample Location**[1]	**Sample ID**[2]	**QC**
WOD/15	Groundwater	15-MW001	WOD-15-MW001-YYYYMMDD	**Arsenic (total and dissolved)**
WOD/15	Groundwater	15-MW007	WOD-15-MW007-YYYYMMDD	**Arsenic (total and dissolved)**
WOD	Groundwater	WOD-MW002D	WOD-MW002D-YYYYMMDD	**Arsenic (total and dissolved)**
Field QC Samples				
QC	Field Duplicate	TBD	WOD-DUP01-YYYYYMMDD	**Arsenic (total and dissolved)**
QC	MS	TBD	Noted on the Chain of Custody as "MS/MSD"	**Arsenic (total and dissolved)**
QC	MSD			**Arsenic (total and dissolved)**

[1] Sample locations are presented in Figures 9-3 and 9-4

[2] YYYYMMDD stands for the year, month, and date the sample is collected. For example, if a groundwater sample is collected from WOD-MW002D on September 25, 2022, the sample ID would be WOD-MW002D-20220925.

[3] One field duplicate, one matrix spike/matrix spike duplicate pair, and one field blank will be collected during each sampling round.

SAP Worksheet # 14: Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2-Worksheet #21)

Field SOP Number	Title	Originating Organization	Equipment Type	Location
SOP-02	Equipment Calibration and Maintenance	Bluestone	Water Level Indicator, Water Quality Meter, PID, Soil Probes	Appendix A
SOP-05	Decontamination of Field Equipment	Bluestone	Decontamination Supplies	Appendix A
SOP-06	Field Documentation	Bluestone	Field Logbook, Field Data Sheets	Appendix A
SOP-10	Storage and Sampling of IDW	Bluestone	UN-approved Drums	Appendix A
SOP-12	Low-Flow GW Sampling and Sampling with Bailer	Bluestone	PID, Water Quality Meter, Groundwater Pump	Appendix A
SOP-13	Sample Packing and Shipping	Bluestone	Coolers, Ice, Bubble Wrap, Tape	Appendix A
SOP-19	Groundwater Elevation Monitoring	Bluestone	Water Level Indicator, Oil/Water Interface Probe	Appendix A

SAP Worksheet #15: Field Equipment Calibration, Maintenance, Testing and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4- Worksheet #22)

Field Equipment	Calibration Activity	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Water Level Meter	NA	NA	Visual Inspection for damages or defects.	Before and after use.	No signs of damage.	Replace.	Field	Manufacturer's instructions.
Water Quality Meter – Horiba, YSI ProDSS or similar	Calibration in accordance with manufacturer's instructions. Verify calibration with standards, use calibration checks to keep meter in range. Have extra calibration fluids and batteries.	NA	Visual Inspection for damages or defects.	Before and after use.	Calibration in range with manufacturer's instructions.	Recalibrate or replace.	Field	Manufacturer's instructions.
Peristaltic Pump	NA	NA	Visual Inspection for damages or defects.	Before and after use.	Pump is operational and maintains flow rates.	Replace.	Field	Manufacturer's instructions.

SAP Worksheet #16: Reference Limits and Evaluation Tables

(UFP-QAPP Manual Section 2.8.1 – Worksheet #15)

Laboratory Limits (Limit of Quantitation (LOQ), Limit of Detection (LOD), and Detection Limit (DL)) are current as of when the information was requested for the SAP and should be considered representative. Concentrations detected between the LOQ and DL will be J-qualified. Non-detects will be reported as the LOD.

The Project Quantitation Limit Goal (PQLG) is PAL/2 (selected in accordance with the Intergovernmental Data Quality Task Force (IDQTF)) QAPP Manual Section 2.6.2.3)

Matrix: Groundwater
Analytical Group: Metals (SW-846 6020A)
Units: µg/L

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference[1]	Project Quantitation Limit Goal (PQLG)	Limit of Quantitation (LOQ)	Detection Limit (DL)
Arsenic	7440-38-2	10.0	EPA National Primary Drinking Water Regulations MCL	5.00	1.00	0.282

[1] PALs determined from the EPA MCL for arsenic as outlined in the WOD ROD.

SAP Worksheet #17: Analytical SOP References

(UFP-QAPP Manual Section 3.2.1 – Worksheet #23)

Andy Johnson, Laboratory PM
Eurofins TestAmerica Pittsburgh
615-301-5045
Andy.Johnson@eurofinset.com

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
PT-IP-003	Acid Digestion of Aqueous Samples, Rev. 15, effective 12/16/2019	Preparation	Aqueous, Metals	N/A	Eurofins TestAmerica Pittsburgh	N
PT-MT-002	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS), Rev 17, effective 9/16/2020	Definitive	Aqueous, Metals	ICP/MS	Eurofins TestAmerica Pittsburgh	N

SAP Worksheet #18: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4 – Worksheet #28)

Matrix: Groundwater
Analytical Group: Metals
Analytical Method/SOP: SW-846 6020A/SOP PT-MT-002
Laboratory Name: Eurofins TestAmerica Pittsburgh

Matrix	Groundwater and aqueous QC samples
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020A/SOP PT-MT-002

Laboratory QC Sample	Number/ Frequency	Method/SOP QC Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Field Duplicate	1 per sampling round	See Worksheet #11	Review sampling procedures. Deviations will be noted during data validation.	Field personnel Data validators	Precision	Same as QC Acceptance Criteria
Field Blank	1 per sampling round	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidance.	Field personnel Data validators	Representativeness (Bias/Contamination)	No target analyte concentrations ≥ 1/2 LOQ.

Matrix	Groundwater and aqueous QC samples
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020A/SOP PT-MT-002

Laboratory QC Sample	Number/ Frequency	Method/SOP QC Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Laboratory Control Sample (LCS)	1 per preparatory batch of up to 20 samples	Arsenic %R = 80 - 120	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Supervisor	Accuracy	Arsenic %R = 84 - 116
Serial Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution results must be within ± 10% from undiluted sample for analytes >50x LOQ	Flag data and comment in Case Narrative.	Supervisor	Accuracy/Bias	Five-fold dilution must agree within ± 10% of the original measurement. Only applicable for samples with concentrations > 50xLOQ (prior to dilution).
Post Digestion Spike (PDS)	One per preparatory batch if MS/MSD fails (using the same sample as used for the MS/MSD if possible).	%R = 80-120	Flag data outside of control limits, and comment in Case Narrative.	Supervisor	Accuracy/Bias	%R = 80-120

Matrix	Groundwater and aqueous QC samples
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020A/SOP PT-MT-002

Laboratory QC Sample	**Number/ Frequency**	**Method/SOP QC Acceptance Criteria**	**Corrective Action**	**Title/Position of Person Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Internal Standards	Every field sample, standard and QC sample.	IS intensity in the samples within 30% - 120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria met. For failed QC samples, correct problem, and rerun all associated failed field samples.	Supervisor	Accuracy	IS intensity in the samples within 30 - 120% of intensity of the IS in the ICAL blank.

Matrix	Groundwater and aqueous QC samples
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020A/SOP PT-MT-002

Laboratory QC Sample	**Number/ Frequency**	**Method/SOP QC Acceptance Criteria**	**Corrective Action**	**Title/Position of Person Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Matrix Spike (MS)	One per preparatory batch.	Use in-house limits. Must contain all surrogates and analytes reported.	Review project specific requirements. Contact client for additional measures. For the specific analyte in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative. For matrix evaluation only. If MS results are outside the limits, the data will be evaluated to determine the source of difference.	Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Groundwater and aqueous QC samples					
Analytical Group	Metals					
Analytical Method/SOP Reference	SW-846 6020A/SOP PT-MT-002					
Laboratory QC Sample	**Number/ Frequency**	**Method/SOP QC Acceptance Criteria**	**Corrective Action**	**Title/Position of Person Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD)	One per preparatory batch.	Use in-house limits. MSD or MD: RPD of all analytes ≤20% (between MS and MSD or sample and MD). MSD: Must contain all surrogates and all analytes to be reported. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the reporting limit.	Examine the project specific requirements. Contact the client for additional measures to be taken. For the specific analytes in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative. The data will be evaluated to determine the source of difference.	Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits

SAP Worksheet #19: Data Verification and Validation Process Table

(UFP-QAPP Manual Section 5.2.1, UFP-QAPP Manual Section 5.2.2, Figure 37 UFP-QAPP Manual, Table 9 UFP-QAPP Manual – Worksheets #34, 35, 36)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Sample log sheets, chain of custody forms, SAP, and laboratory sample login documentation	The FOL will verify that samples were correctly identified, chain of custody records are legible, data will be traceable to the corresponding samples, the samples listed in **Worksheets #11 and #13** were collected from intended locations, and the correct sampling and analytical methods/SOPs were assigned to samples listed on the chain of custody record. The PM will verify that the sampling plan was implemented and carried out as written and will make sure that any significant deviations are documented in the project report.	FOL and PM, Bluestone	I	Internal
Chain-of-custody forms	Verify that the chain-of-custody form is complete and accurate; and was signed and dated by the sampler relinquishing the samples and by the laboratory receiving the samples. Resolve discrepancies, if possible. Alert the Bluestone PM verbally or via email if discrepancies are unresolvable.	FOL, Bluestone	I	Internal
Chain-of-custody forms	Verify sample shipment completeness against the chain-of-custody record, verify proper sample preservation/integrity, sign to indicate receipt, note any discrepancies, and correct them as necessary. Notify the Bluestone FOL or PM of any deviations from sample shipping requirements such as damaged sample containers, or inappropriate temperature or pH. Note uncorrectable discrepancies in the data package case narrative.	Laboratory sample custodian, Eurofins/TestAmerica	I	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Analytical calibration standards	Verify that standards are traceable and meet contract, method, and procedural requirements, as applicable, and include certificates of analysis in the laboratory data package to document the traceability. If discrepancies in traceability are found, bring the discrepancies to the laboratory PM attention for correction.	Laboratory analyst, Eurofins/TestAmerica	I	Internal
SAP, analytical SOPs, and analytical data packages	Verify that the correct analytical methods/SOPs were applied. Establish that method QC samples were analyzed and in control as listed in the analytical SOPs. If method QC is not in control, the Eurofins/TestAmerica PM will contact the Bluestone Project Chemist or PM verbally or via email for guidance prior to laboratory data package preparation.	Laboratory PM, Eurofins/TestAmerica	I	Internal
Laboratory analytical data package	Verify the analytical data package for completeness and accuracy, including certificates of analysis for calibration and check standards. The laboratory QAM will sign the case narrative for each data package.	Laboratory QAM, Eurofins/TestAmerica	I	Internal
Laboratory analytical data package	Review chain-of-custody records to ensure that the required analytical samples were collected, appropriate sample identifications were used, and correct analytical methods were applied to each sample. Verify the analytical data package for completeness and accuracy, including certificates of analysis for calibration and check standards. Obtain missing data package elements from the laboratory. Document unrecoverable elements, if any, in the data validation report submitted to the Bluestone PM and alert the project chemist or PM.	Data validator, Bluestone	I/IIa	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
EDDs/Analytical data packages	Verify a representative subset of EDD results for accuracy and completeness against the data package and verify the chain of custody records at the start of validation. If required elements are missing, obtain missing elements form the laboratory before completing the validation. If any element cannot be obtained, document the omission in the DV report and identify the missing elements to the Bluestone project chemist or PM as early as possible.	Data validator, Eurofins/TestAmerica	I/IIa	External
Sample shipment and storage conditions; and holding times for representativeness	Verify that sample shipping and storage conditions satisfy **Worksheet #13** requirements. Document deviations from requirements in the DV report and notify the Bluestone project chemist or PM if deviations from the SAP requirements are serious enough to warrant data rejection. Document findings in the DV report.	Data validator, Bluestone	I/IIa	External
QC samples/MPC compliance	Ensure that the scheduled laboratory and field QC samples were submitted for analysis and that the MPCs listed in SAP **Worksheets #11, #16, and #18** were met for all field samples and QC samples. Document findings in the DV report. Evaluate sample results for laboratory contamination and qualify false detections using the laboratory method/preparation blank summaries. Qualify analyte concentrations between the MDL and the reporting limit as estimated ("*J*" qualifier). Replace laboratory flags with validation qualifiers on validated data in accordance with the laboratory data validation process described below and document findings in the data validation report. Retain laboratory flags in the EDD to document data as received from the laboratory.	Data validator, Bluestone	IIa/IIb	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Field and laboratory duplicate analyses for precision	Verify field sampling precision by checking RPDs for field duplicate samples. Verify laboratory precision by checking RPDs or %D values from calibrations, laboratory duplicates, MS/MSDs, and LCS/laboratory control sample duplicates (LCSDs). Ensure compliance with MPC accuracy and precision goals listed in **Worksheets #11 and #18**. Document findings in the DV report.	Data validator, Bluestone	I/IIb	External
SAP/Laboratory data packages/ EDDs	Conduct EPA Stage 4 data validation on 100% of the definitive laboratory data generated by the selected methods using QC criteria listed in this SAP. Apply validation qualifiers in accordance with logic provided in the National Functional Guidelines (NFG) for Inorganic Superfund (EPA, 2020a) data review (including EPA Region-specific requirements, if applicable). Document findings in the DV report.	Data validator, Bluestone	IIa/IIb	External

Usability Assessment

(UFP-QAPP Manual Section 2.6.2 and 5.2.3 Worksheet #37)

After data validation, project team members designated by the Bluestone PM will assess whether the data collectively can be used as intended to support the attainment of project objectives. They will consider whether any missing or rejected data, deviations, or corrective actions have compromised the ability to make decisions or to make the decisions with the desired level of confidence.

The following data quality indicators (DQIs) will be reviewed during the data usability assessment:

Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Precision describes the reproducibility of measurements of the parameter for samples analyzed under similar conditions. Precision is expressed as a range (the difference between two measurements of the same parameter) or as Relative Percent Difference (RPD); the range relative to the mean, expressed as a percent.

RPD will be calculated as follows:

$$\text{RPD} = \frac{|\text{amount in Sample 1} - \text{amount in Sample 2}|}{0.5\,(\text{amount in Sample 1} + \text{amount in Sample 2})} \times 100$$

Field precision will be assessed by the collection and analysis of field duplicate samples and precision will be expressed as the RPD. Laboratory precision will be assessed by calculating RPDs for laboratory duplicates, laboratory control sample duplicates, or matrix spike duplicates (MSDs). Results of all laboratory and field duplicates will be evaluated based on the measurement performance criteria presented on Worksheets #11 and #18. Results outside the precision limits will trigger corrective action.

Accuracy

Accuracy is the degree of agreement between the observed value and an accepted reference value. Accuracy is expressed as a percent recovery (%R). Accuracy measurements are designed to detect biases resulting from sample handling and analysis processes.

The %R for each spiked sample will be calculated as follows:

$$\%R = \frac{|\text{Amount in Spiked Sample} - \text{Amount in Sample}|}{\text{Known Amount Added}} \times 100$$

The %R for LCSs will be calculated as follows:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \times 100$$

Laboratory accuracy, expressed as %R, will be determined through the analysis of matrix spike and matrix spike duplicate samples (MS/MSD), and laboratory control (LCS) samples. MS analyses measure the combined accuracy effects of the sample matrix, sample preparation, and sample measurement. LCSs are used to assess the accuracy of laboratory operations with minimal sample matrix effects. The achievement of accurate data in the field will be addressed through the use of procedures that minimize bias, including calibration of field instruments and adherence to sample holding times and preservation requirements. Accuracy in the field will be evaluated through the use of field blanks to detect cross-contamination. Results for all laboratory method blanks, equipment blanks, MS/MSD, and LCS samples will be evaluated. The results for each analyte will be checked against the measurement performance criteria presented on Worksheets #11 and #18.

Sensitivity

Sensitivity is the ability of the laboratory method or instrument to detect the target analytes at the level of interest. The appropriate analytical methods will be selected to achieve LOQs that are sufficiently below the cleanup levels. Sensitivity of the laboratory-specific LOQs to clean-up levels for LTM will be evaluated before the laboratory is selected.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population or an environmental condition at the site. Representativeness is a qualitative parameter that is dependent on the proper design of the sampling program and proper laboratory protocol. The rationale of the sampling program is discussed in this SAP. Ensuring that the SAP is followed, proper sampling techniques are used, proper analytical procedures are followed, and sample holding times are not exceeded in the laboratory will satisfy representativeness.

Comparability

Comparability expresses the confidence with which one data set can be compared to another (e.g., between sampling points, between sampling events). The extent to which existing and planned data will be comparable depends on the similarity of sampling techniques and analytical methods. The procedures used to obtain the planned data, as documented in this SAP, are expected to provide comparable data. Any changes in procedures or QA objectives, however, may affect data comparability. Results will be reported in consistent units of measure and reporting and detection limits that ensure comparability with previous data.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. “Normal conditions” are defined as the conditions expected if the sampling plan were implemented as planned.

A completeness check will be done on all data generated. Field and analytical completeness will be calculated, with a goal of 95% for each value.

The field completeness will be calculated by the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in this document. The equation for field completeness is:

$$\%\ Field\ Completeness = \frac{Number\ of\ Samples\ Received\ by\ Laboratories}{Total\ Number\ of\ Samples\ Planned\ to\ be\ Collected} \times 100$$

The analytical completeness will be calculated by the ratio of total valid analytical data results (including estimated values) to the total number of analytical results requested on samples submitted for analysis. Valid analytical data results are defined as those that were not rejected during data validation, due to a significant quality assurance/quality control problem. The equation for analytical completeness is:

$$\%\ Analytical\,Completeness = \frac{Total\,Valid\ Analytical\,Data}{Analytical\,Data\,Obtained}\, x\,100$$

SAP Worksheet #20 References

Data Summary Report, July 2020 Groundwater Monitoring, Waste Oil Dump, NASA Wallops Flight Facility, LJT & Associates Inc., February 2021.

Feasibility Study, Waste Oil Dump, NASA Wallops Flight Facility, Wallops Island, Virginia. TtNUS, October 2005.

Long Term Monitoring Plan Rev 3 - Waste Oil Dump Site, Wallops Island, Virginia. Tetra Tech, Inc., September 2015.

Long Term Monitoring Plan, Waste Oil Dump Site, NASA Wallops Flight Facility, Virginia. TtNUS, October 2009b.

Pilot Study Report, Waste Oil Dump Site. NASA Wallops Flight Facility, Virginia. TtNUS, March 2009a.

Preliminary Remedial Action Plan, Waste Oil Dump Site. NASA Wallops Flight Facility, Wallops Island, Virginia. TtNUS, December 2007.

Record of Decision, Waste Oil Dump, NASA Wallops Flight Facility, Wallops Island, Virginia. TtNUS, March 2008.

Remedial Action Completion Report, Waste Oil Dump, NASA Wallops Flight Facility, Wallops Island Virginia. TtNUS, April 2011.

Supplemental Remedial Investigation Report, Waste Oil Dump, NASA Wallops Flight Facility, Wallops Island, Virginia, TtNUS, December 2004.

Wallops Flight Facility Site-Wide Programmatic Environmental Impact Statement, NASA, May 2018.

Appendix A
Bluestone SOPs

Standard Operating Procedure Number 02
EQUIPMENT CALIBRATION AND MAINTENANCE

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for performing routine calibrations of equipment used in environmental investigations. This SOP also includes necessary steps for equipment long-term care and maintenance.

[NOTE: Bluestone SOP Number 2 incorporates a project-specific equipment list.]

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Site Safety and Health Plan (SSHP), the Bluestone Corporate Safety and Health Program, and site and/or client-specific requirements. Potential health and safety hazards associated calibration and equipment maintenance include splashing from calibration fluids and operating compressed gas cylinders, and gas fumes. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for equipment calibration and maintenance, and includes safety glasses, safety shoes and nitrile gloves. Additional details regarding site safety and health will be provided in the APP and/or SSHP.

4.0 PROJECT-SPECIFIC EQUIPMENT

4.1 Water Level Indicator

Procedures for the maintenance of a water level indicator is described in the following sections. The operation manual supplied by the manufacturer will be reviewed for instructions concerning the variations pertinent to specific equipment brands and models.

4.1.1 Preventative Maintenance

The water level indicator will be rinsed with soapy water, followed by deionized (D)I water after each use to avoid cross contamination between groundwater wells. The probe will be kept free of silt and product coatings. Dye-free paper towels will be used to wipe the probe.

4.2 Water Quality Meter – YSI Pro Plus (or similar model)

The procedures for calibration and the O&M of the YSI Pro Plus multiparameter meter is described in the following sections. This meter may be used to measure pH, specific conductance, temperature, turbidity, oxidation-reduction potential (ORP), and dissolved oxygen (DO). The operation manual supplied by the manufacturer will be consulted for instructions concerning the operation for other water quality parameters.

4.2.1 Calibration

Calibration will be performed each day prior to sample collection activities or when variability is noted. The YSI Pro Plus requires a one-point calibration for specific conductance and ORP, a two-point calibration for turbidity and a three-point calibration for pH. A separate calibration solution is required for most of the parameters, although one solution may be used to calibrate multiple parameters (that is, a 7 pH and 0 NTU turbidity solution). Each parameter will be calibrated on an individual basis as outlined in the instruction manual.

4.2.2 Preventative Maintenance

The YSI Pro Plus meter will be cleaned and inspected daily before and after use. The batteries will be replaced frequently, and the electrodes will be replaced when cracked or when the instrument cannot maintain a calibration to the manufacturer specifications. The pH sensor must be kept moist by filling the small rubber cap with water and storing the electrode with the cap attached.

4.3 Photo-Ionization Detector (MiniRAE® 3000, or similar model) & Multi-Gas Meter (MultiRAE®, or similar model)

Procedures for the calibration and O&M of a MiniRAE® 3000 photo-ionization detection (PID) and a MultiRAE® multi-gas meter are described in the following sections. At a minimum, these meters will be calibrated daily; however, more frequent calibrations may be necessary if the instrument begins to drift from background (0 parts per million [ppm]). Justification and rationale for performing additional calibrations will be noted in the field book. Directions for calibrating are listed below and are available in the operation manual supplied by the manufacturer.

4.3.1 Calibration

To calibrate the MiniRAE® PID meter:

1. Activate the MiniRAE® unit by pressing the [MODE] key.
2. The unit will perform a self-diagnostic routine.
3. After the MiniRAE® has gone through its self-diagnostic routine, simultaneously press [N/-] and [MODE] keys for three seconds. This will put the meter in Programming Mode.
4. The meter will prompt “Calibrate /Select Gas”. Press [Y/+] key.
5. The meter will prompt “Fresh air cal?” Press [Y/+] key. The display shows “zero in progress” followed by “wait...” and a countdown timer.
6. After about 15 seconds, the display will show the message “update data…zeroed…reading = X.X ppm…”. Record this reading in the field book or calibration log sheet. Note: Make sure the Fresh Air Cal is done outside because this is the value the meter will use as zero.
7. The meter will prompt “Span cal?’ Press [Y/+] key.
8. The meter will prompt if the cal gas is Isobutylene. Press [Y/+] key.
9. The meter will then ask the user to “Apply gas now!” Note: Make sure the meter is connected to the Isobutylene canister and open the pressure valve to release the gas.

10. The display will show "wait…30" with a countdown timer showing the number of remaining seconds. When the countdown timer reaches 0, the display shows the calibrated value. Record this value in the field book or calibration log sheet.
11. The reading should be close to the actual concentration of the gas. If not, wait a few seconds and press the [Mode] key. This process may need to be repeated until the calibration gas as stabilized to 1 to 2 parts per million (ppm) with the calibration gas range.
12. Press the [MODE] key to exit the standard gas calibration mode, turn off the gas, and disconnect the calibration gas from the MiniRAE®. The calibration gas used for calibration is isobutylene at a concentration of 100 ppm. The use of this calibration gas will result in a reading of 100 ppm during the calibration mode.

4.3.2 Preventative Maintenance

The MiniRAE® unit will be charged at the end of every workday.

[NOTE: Due to instrument complexity, the use of a hand-held Global Positioning Survey (GPS) unit is discussed separately in Bluestone's SOP Number 04 – *GPS Survey*.]

4.4 Direct Soil pH Probe

Procedures for the calibration and maintenance of a direct soil pH probe are described in the following sections. The operation manual supplied by the manufacturer will be reviewed for instructions concerning the variations pertinent to specific equipment brands and models.

4.4.1 Calibration

Calibration will be performed each day prior to sample collection activities or when variability is noted. The Hanna Instruments® 99121 (HI 99121) model (or similar) will be calibrated using two calibration buffers for pH, first using pH 7 buffer followed by pH 4 buffer.

4.4.2 Preventative Maintenance

The HI 99121 will be cleaned and inspected daily before and after use. The battery life indicator will be inspected daily, and batteries will be replaced if the battery life is low. The electrode will be inspected before use for scratches and cracks, and to ensure the reference junctions are flowing. Any salt deposits will be rinsed with water. The electrolyte filling solution (3.5M KCl with AgCl reference electrolyte) will be topped off, as necessary. The electrode will be cleaned by soaking with vendor-supplied cleaning solution for 20 minutes each day to remove soil deposits.

4.5 Soil ORP Probe

Procedures for the calibration, maintenance, sample preparation, and sample calculations associated with a dedicated soil ORP probe are described in the following sections. The operation manual supplied by the manufacturer will be reviewed for instructions concerning the variations pertinent to specific equipment brands and models.

4.5.1 Calibration

Calibration will be performed each day prior to sample collection activities or when variability is noted. The Hach® Pocket Pro ORP model (or similar) will be calibrated with a 1-point calibration. When the measurement is stable, the displayed ORP value will be adjusted until it is equivalent with the calibration solution.

4.5.2 Preventative Maintenance

The Hach® Pocket Pro will be cleaned and inspected daily before and after use. The sensor will be cleaned when stabilization is slow, or results drift or are not accurate. The sensor will be soaked in vendor-supplied cleaning agent and risked with deionized water for 1 minute. The battery life indicator will be inspected daily, and batteries will be replaced if the battery life is low.

4.5.3 Sample Preparation

Soil reduction potential (Eh) can be measured by creating a soil suspension with the ratio of one part soil to 2 parts distilled water (e.g., 50 g of soil to 100 mL of water). The sample should be sufficient to fully immerse the sensor. The slurry should be well mixed with a stirring tool.

4.5.4 Sample Calculations

The voltage obtained as ORP readings can be converted to Eh by correcting for the electrode potential of the reference electrode. Generally, reference values for electrodes range from +236 mV for 1M KCL to +197 mV for saturated KCl solutions. The Hach® ORP Standard Solution is 220 mV; therefore, to convert ORP readings collected with the Hach® to Eh, 220 mV will be added to the ORP voltage.

5.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP Number 05 – *Decontamination of Field Equipment*.

6.0 DATA MANAGEMENT

Equipment calibration values will be recorded in the field logbook and on field data sheets, as necessary. Bluestone's SOP Number 06 – *Field Documentation* details the methods for data collection and management, and additional information will be specified in the site-specific Work Plan.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel will read and acknowledge the site-specific Work Plan, UFP-QAPP, and any applicable SOPs.

Standard Operating Procedure Number 05
DECONTAMINATION OF FIELD EQUIPMENT

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology used to perform decontamination activities during an environmental investigation. All sampling and investigation equipment that comes in contact with soil or groundwater must be decontaminated prior to use in the field, between soil borings, between sampling locations, and at the completion of field activities.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Site-Specific Health and Safety Plan (SSHP), the Bluestone Corporate Safety and Health Program, and site and/or client-specific requirements. Potential health and safety hazards associated decontamination activities include contact with potentially impacted media, splashing of decontamination fluids, pinch points, and slip/trip/fall hazards, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with decontamination tasks, and includes, safety glasses, safety shoes and nitrile gloves. Additional details regarding site safety and health must be provided in the APP and/or SSHP.

4.0 DECONTAMINATION EQUIPMENT

Standard equipment and supplies necessary to perform decontamination:

- De-ionized water
- Potable water
- Spray bottle
- 5-gallon pail
- Scrub brush
- Non-phosphate laboratory-grade detergent
- Plastic sheeting
- Contractor bags
- Nitrile gloves
- Paper towels

5.0 EQUIPMENT-SPECIFIC DECONTAMINATION PROCEDURES

5.1 Decontamination of Non-Dedicated Pumps

Well pumps will be decontaminated using to the following procedure:

1. Disassemble the pump and place on clean plastic sheeting. Note, plastic sheeting must be replaced between each location.
2. Use a distilled water and non-phosphate laboratory-grade detergent mixture to spray down the inner and outer pump and use a scrub brush to wipe away any visual sediment or residual contaminants.
3. Rinse all pieces of the pump with DI water twice before reassembling. Containerize all decontamination water in a bucket.
4. Place the equipment on clean plastic and allow it to air dry.
5. Wrap equipment in plastic or aluminum foil to store until next use.
6. Retain decontamination fluids for disposal as described in the Bluestone SOP Number 10 – *Storage and Sampling of IDW*.

5.2 Decontamination of Sampling Equipment

Non-disposable and other non-dedicated equipment used to collect soil and/or groundwater samples will be decontaminated prior to use. This equipment includes, but is not limited to, stainless-steel knives and spoons, split-spoon barrels, direct-push shoes and rods, and stainless-steel bowls used to homogenize soil samples.

Sampling equipment will be decontaminated using the following procedures:

1. Fill a non-metallic wash tub or bucket with approximately 6 inches of DI water. Mix a detergent solution in the tub. The solution shall consist of approximately 1 tablespoon of non-phosphate laboratory-grade detergent (e.g. Liquinox®) per gallon of water.
2. Scrub all sampling equipment with a stiff-bristled brush and detergent solution.
3. Transfer the equipment to a separate tub partially filled with DI water and thoroughly rinse each piece of equipment.
4. Perform a second rinse using fresh DI water and collect fluid into the dedicated rinse tub.
5. Place the equipment on clean plastic sheeting and allow it to air dry.
6. Wrap equipment in plastic or aluminum foil to store until next use. Retain decontamination fluids for disposal as described in Bluestone's SOP Number 05 – *Decontamination of Field Equipment*.

5.3 Decontamination of Drilling Equipment and Heavy Machinery

Drilling tools including drill string, augers, drill bits, direct-push rods, etc., in addition to heavy equipment (i.e., drill rigs, excavators, etc.) will be decontaminated between each borehole according to the following procedures:

1. Construct a decontamination pad using heavy plastic sheeting other waterproof materials or use a decontamination trailer or tank.
2. Back the drill rig or direct-push rig into the decontamination pad/trailer/tank or place equipment in a rack off the ground inside the unit, as appropriate.
3. Remove all visible soil and contamination from equipment surfaces by steam cleaning.

Include the inside of drill string, augers, and direct-push rods. If necessary, use a stiff-bristled brush to physically remove residual soils and contamination.

4. Transport the unit to an area free of IDW and known impacts and allow the equipment to air dry.
7. Retain all decontamination fluids for disposal as described in Bluestone's SOP Number 10 – *Storage and Sampling of IDW*.

6.0 EQUIPMENT BLANK SAMPLING

For quality control purposes, an equipment blank sample must be collected if analytical samples are being collected. Equipment blanks are typically collected at a frequency of one per every 10 to 20 field samples. Equipment blank samples are collected by pouring a volume of laboratory-prepared DI water over a freshly decontaminated piece of equipment and into the sample bottle to determine the effectiveness of decontamination procedures. The equipment blank sample will be collected after sampling in contaminated areas, rather than after sampling background areas.

7.0 DATA MANAGEMENT

Pertinent information obtained during decontamination activities (i.e., times and methods used) will be recorded in the field logbook. Bluestone's SOP Number 06 – *Field Documentation* details the methods for data collection and management, and additional information will be specified in the Work Plan. Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photographs must be in accordance with contract requirements and site-specific security requirements.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the site-specific Work Plan, UFP-QAPP, and any applicable SOPs.

Standard Operating Procedure Number 06
FIELD DOCUMENTATION

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methods for recording and managing data obtained by performing tasks associated with an environmental investigation and/or long-term monitoring activities. Data management is a critical part of any field investigation, and requires diligence in maintaining accuracy, organization, and confidentiality.

Every sample, field measurement and investigative activity conducted during a project must be properly documented using the official field logbook or approved field data sheets including, but not limited to, geologic logs, well construction logs, soil boring logs, calibration forms, field sampling forms, and health and safety tailgate sheets. The use of electronic field data sheets must be consistent with contract requirements and the site-specific Work Plan. Field documentation must be adequate to support each step of the investigation, from the point of sample collection to final validated data reporting. Records must be available to identify, track and monitor individual samples and provide details for all investigative activities.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

2.1 Field Logbooks

Information pertinent to an investigation or long-term monitoring must be recorded in a bound logbook with consecutively numbered, water-resistant pages. All site activities must be recorded in the field logbook including, but not limited to, arrival and departure times, a chain of events, progression of work tasks and a list of site visitors. The field personnel responsible for the entries will sign and date each entry or page. All logbook entries will be made in indelible ink. The time and date of each entry will be noted in the logbook.

Field logbooks must be kept in the field personnel's possession or in a secure location during field operations. Following the investigation or monitoring activities, logbooks will become part of the project file, and will serve as the official site document in the event of legal action. The following list contains typical field logbook entries:

- Date
- Weather conditions
- Client
- Names of all field personnel, including subcontractors
- Site name, municipality, and State
- Location of samples (may include a sketch)
- Sample media

- Time (military) of sample collection
- Sample nomenclature
- Interval and depth of sample
- Sample collection methods
- Sample physical description
- Field observations
- Quality Control (QC) sample information
- Number assigned to chain-of-custody (COC)

In addition to pertinent sampling information, details with respect to anticipated health and safety hazards, required personal protective equipment (PPE) and a record of daily field safety briefings will be included in the field logbook.

2.2 Field Data Sheets

Sampling data will be recorded on either paper or electronic Field Sample Data Sheets. The following information will be recorded:

- Sample ID
- Sample Location
- Sample Date/Time
- Sample Collection Method
- Sample Description
- Sample Type
- Sampled By

3.0 PHOTOGRAPHIC DOCUMENTATION

Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photographs must be in accordance with contract requirements and site-specific security requirements. Project photographs may include the sample, sample collection activities, surrounding areas, disturbances caused by access of heavy equipment or intrusive activities, and final site restoration. Photographs taken to document sampling points will include two or more reference points to facilitate a potential return to the sample point, if necessary.

4.0 SAMPLE DOCUMENTATION

4.1 Sample Nomenclature

In accordance with the site-specific Work Plan and/or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP), each sample will be assigned a unique sample identification number that appears on all sample labels, COCs, field logbooks, and all other applicable documentation forms.

The sample nomenclature typically consists of three to four alphanumeric code segments, each separated by hyphens. The sample tracking number identifies the site, sample type or medium, location, depth (for soil samples), and sampling date. QC sample designations will be added, as appropriate, following the nomenclature presented below. However, the Work Plan and/or UFP-QAPP will be consulted to determine the if there any site-specific requirements. Other pertinent information regarding sample identification will be recorded in the field logbooks or on sample log sheets. The sample numbers will be in the following format: "AA-BBCCC_QEEEE_QC Indicator".

The sample tracking number format is designated as follows:

1) Location ID

AA: Area of Concern (AOC) identifier (AOC 1 = 01; AOC 3 = 03, etc.)

BB: Sample Media, including:

SS: Surface Soil

BS/BD: Subsurface Soil [S=shallow (1-5 ft bgs); D=deep (5-10 ft bgs)]

GW: Groundwater

PW: Pore water

2) Unique timeframe indicator

Q: Quarter identified (First Quarter = 1Q; Second Quarter = 2Q)

EEEE: Year (Example = 2019)

3) QC indicator (if applicable)

DUP: Field duplicate samples will be collected with 'DUP' in the 'AOC identifier'. The actual location of the duplicate sample will be noted in the field logbook.

MS: Matrix spike samples will be labeled with the standard sample convention, followed by 'MS' to clearly indicate to which sample the spike sample correlates.

MSD: Matrix spike duplicate (MSD) samples will be labeled with the standard sample convention, followed by 'MSD' to clearly indicate to which sample the spike sample correlates.

4.2 CCC Sequential Sample Location Number (001, 002, Etc.) Sample Labels and Tags

Each sample collected at a site and transported to a laboratory for analysis will be identified by a sample label, with specific information regarding the sample. Completed sticker labels must be securely attached to the sample container and include the following information:

- Date
- Time (military) of sample collection
- Type of analyses requested
- Sample number
- Sample collection depth
- Location of sample collection

- Type of preservative
- Initials of sampler

4.3 Chain-of-Custody Records

The COC serves as physical evidence of sample custody over the life of the sample batch. Field personnel will initiate a COC at the time of sample collection. All custody transfers of the sample batch will be recorded on the COC by the individual relinquishing and the receiver of the samples, and signed, dated, and time stamped at the time of transfer. Each cooler is assigned a separate COC on which only the samples packed in that cooler are listed.

Laboratory-specific COCs will be provided and in general include the following information:

- Sample identification numbers
- Signature(s) of field personnel
- Date of collection
- Time (military) of collection
- Sample type (solid, liquid, etc.)
- Sample ID
- Number of containers per sample
- Preservative(s) used
- Requested analytes
- Signatures of all sample handlers
- Inclusive dates and times of possession
- Description of compromised sample integrity (if applicable)
- Temperature of cooler upon receipt by laboratory

After completing the COC, the original will be enclosed in a plastic bag and taped to the inside lid of the cooler.

4.4 Custody Seals

Custody seals will be placed over the lid of the cooler and remain in place from the time the coolers are packed until they are opened by laboratory personnel in order to preserve the integrity of the cooler during shipment. Custody seals must be attached so that it is necessary to break the seals to open the cooler. The custody seals will be secured with clear tape. As long as the COCs are sealed inside the sample cooler and custody seals remain intact, laboratory couriers are not required to sign the COC.

5.0 DAILY REPORT

A daily report will be generated at the end of each sampling day, and will include the following information:

- Date
- Prepared by

- Project and Site name
- Weather conditions
- Level of PPE
- Description of project activities
- Employees and sub-contractors present on site
- Documentation of tailgate safety meetings
- Daily equipment and material deliveries
- Material(s) shipped off site
- List of samples collected
- Submittals
- Planned activities for the next day

6.0 CORRECTIONS TO DOCUMENTATION

Data entries will be recorded with indelible ink. Accountable serialized documents will not be destroyed or discarded, even if they are illegible or contain inaccuracies that require a replacement document. Errors will be corrected by marking a single line through the error, entering the correct information, and initialing and dating the correction. The erroneous information will not be whited out. Any subsequent errors discovered later will be corrected, initialed, and dated by the individual who made the original entry.

7.0 COMMUNICATION AND TECHNICAL DIRECTION

Field personnel will support one another and maintain open communication during all aspects of the field activities. When a technical point is in question, field personnel will communicate with the Project Manager (PM) or technical lead for clarification and/or additional direction. Deviations, exceptions, and/or omissions from the Work Plan or best management practices must be communicated to the Project Manager in a timely manner. Maintain records of all such communications in a field logbook, describing the issue, the outcome, and individuals involved in the decision.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the site-specific Work Plan, UFP-QAPP, and any applicable SOPs.

Standard Operating Procedure Number 10
STORAGE AND SAMPLING OF INVESTIGATION-DERIVED WASTES (IDW)

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for characterization and handling of investigation-derived wastes (IDW). A field investigation typically generates solid and/or liquid wastes to be stored and characterized for disposal purposes. This material is classified as IDW, and typically includes soil cuttings generated through soil borings and monitoring well installations, groundwater sampling purge water, and associated decontamination fluids generated during the investigation or long-term monitoring activities.

Waste management procedures for IDW are based on the requirements specified in Title 40 of the CFR, Part 262 (40 CFR 262) *Standards Applicable to Generators of Hazardous Waste* and industry best management practices. This SOP describes the proper on-site disposal, containerization, labeling, and storage of solid and liquid IDWs. Site-specific IDW disposal requirements will be detailed in the Work Plan, Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) and/or Accident Prevention Plan (APP).

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Site Safety and Health Plan (SSHP), the Bluestone Corporate Safety and Health Program, and site and/or client-specific requirements. Potential health and safety hazards associated with waste characterization and disposal include exposure to contaminants via direct contact, inhalation or splashing, heavy lifting, pinch points, and slip/trip/fall hazards. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with IDW sampling tasks, and includes a safety glasses, safety shoes, leather and/or nitrile gloves, and high-visibility vests. Additional details regarding site safety and health will be provided in the APP and/or SSHP.

4.0 IDW CHARACTERIZATION

4.1 Liquid IDW

Liquid IDW includes well development water, decontamination fluids, and groundwater monitoring purge waters. Liquid IDW may be characterized using associated groundwater quality data generated from the respective samples or direct sampling, and typically qualifies as non-hazardous waste. Liquid IDW may be stored in a bulk frac tank or containerized separately in drums.

On-site disposition of liquid IDW is acceptable in some states, provided the wastes qualify as non-hazardous and do not pose a threat to surrounding properties. All liquid waste known or suspected to be hazardous waste must be contained. Drums and polyethylene totes may be used for interim storage and transport of liquid IDW. Labels will be prepared in accordance with applicable regulations and the guidelines outlined below. Containers must be kept closed and secured except when filling or disposing of the contents. Manufacturer Department of Transportation (DOT) specifications will be followed when sealing containers in preparation for transport.

United Nations (UN)-approved drums (49 CFR 173.3), polyethylene tanks, and 5-gallon pails may be used to collect liquid IDW, as task appropriate. Liquid IDW collected in 5-gallon pails will be transferred to drums or totes as soon as possible after collection. Hazardous (or suspected) liquid IDW must be containerized in UN-approved drums.

Containers of liquid IDW will be labeled to indicate the source and nature of the waste material. The following information must be visible on the top or sides of each container: container number(s), facility name, associated monitoring well or borehole ID, date of generation, container contents, estimated quantity, and the client point of contact (POC).

Containers will be marked with 2-inch letters and numbers using a waterproof paint pen. A complete inventory of the IDW will be maintained by the Field Team Leader to facilitate identification and tracking of liquid IDW for appropriate disposal. This inventory will document the information listed above, in addition to the location of the container, and initials of the responsible POC. The total number of containers of liquid IDW generated will be recorded in the field logbook at the end of each workday. Containers of liquid IDW characterized as hazardous or non-hazardous transported off-site for disposal will be labeled in accordance with applicable State and Federal requirements including, but not limited to, Resource Conservation and Recovery Act (RCRA) guidelines, the Toxic Substances Control Act (TSCA), and DOT (40 CFR 171-179).

Containers of liquid IDW will be staged temporarily on site until characterization is complete. Containers of liquid IDW stored during winter months will be under-filled to allow for expansion during freezing. IDW will be stored in secure areas of the site, where containers are protected from flooding, traffic, and unauthorized access or tampering.

For IDW pending characterization, all containers must be properly sealed and labeled, and may be staged on pallets until characterization is complete. If possible, IDW containers will be secured with temporary chain-link fencing; however, caution tape and/or temporary orange construction fencing may also be used as need to protect the IDW containers.

Containers of liquid IDW will be characterized and disposed of accordingly in a timely manner. If liquid-filled containers remain in storage for 30 days or more, the containers must be staged on polyethylene sheeting that is surrounded by a retention berm (i.e. 2x4 lumber), and positioned with enough separation so all sides of the containers can be monitored for leaks.

4.2 Solid IDW

Solid IDW includes soil cuttings from sources such as, but not limited to, direct-push sampling, soil borings, and installation of monitoring wells. Solid IDW must be containerized and staged pending proper characterization. To characterize solid IDW, a composite sample consisting of aliquots from each container will be submitted for analysis. In addition, the quality of solid IDW will be evaluated based on data generated from primary sample and historical site data.

Expended personal protective equipment (PPE) may also be considered IDW, depending on site conditions and/or state regulations, in addition to disposable sampling equipment (i.e., bailers, string, acetate liners, etc.), depending on the nature of site contaminants. In most cases, expended PPE and sampling equipment may be double bagged and disposed with other municipal waste at a local sanitary landfill. All gross contamination removed from the PPE and disposable equipment will be added to the appropriate IDW drums/totes.

Solid IDW consisting of soil cuttings and excess soil volume, will be placed in a lined roll-off dumpster or in UN-approved drums. Five-gallon pails may be used for interim handling and transport of solid IDW. On-site disposition of solid IDW (soil cuttings and excess soil volume) may be appropriate, provided the material is characterized as non-hazardous and such disposition is acceptable under State and local regulations. Any soils suspected of being hazardous must be drummed rather than placed into a roll-off dumpster. Containers must be kept closed and sealed except when adding to or disposing of the contents. Manufacturer DOT specifications must be followed when sealing containers in preparation for transport.

Based on the size of the site, each area of concern (AOC) may require a staging area for solid IDW. Each staging area will be characterized separately, in accordance with the *Management of Investigation Derived Waste During Site Inspections*, EPA/540/G-91/009 (USEPA, 1991). However, solid IDW generated from multiple soil borings within a single AOC may be characterized as a single waste stream.

Containers of solid IDW must be labeled to indicate the source and nature of the waste media. The following information will be marked on the top or sides of each container: container number(s), site name, associated monitoring well or soil boring ID, borehole number, date of generation, container contents, estimated quantity, and the client POC.

Containers will be marked with 2-inch letters and numbers using a waterproof paint pen. A complete inventory of IDW will be maintained by the Field Team Leader to facilitate identification and tracking of solid IDW for characterization and disposal. This inventory will document the details list above, in addition to the location of the container, and initials of the responsible POC. The IDW inventory and the total number of containers of solid IDW generated will be recorded in the field logbook at the end of each workday.

Solid IDW characterized as hazardous (based on laboratory analytical results) will be re-labeled in accordance with applicable State and Federal requirements including, but not limited to, RCRA, TSCA, and DOT. Containers of solid IDW will be staged temporarily at the site until characterization is complete. Solid IDW will be stored in secure areas of the site, where containers are protected from flooding, traffic, and unauthorized access or tampering. Secondary containment structures will be implemented as required for the storage of solid IDW.

For solid IDW pending characterization, all containers must be properly sealed and labeled, and may be staged on pallets until characterization is complete. If possible, IDW containers will be secured within temporary chain-link fencing; however, caution tape and/or temporary orange construction fencing may also be used as need to protect the IDW containers. Containers of solid IDW will be characterized and disposed of accordingly in a timely manner.

5.0 IDW CHARACTERIZATION SAMPLING

The sampling procedures for liquid and solid IDW contained in drums are described in the following sections. Within two weeks of the completion of field activities, an aliquot from each waste stream will be collected and composited except for samples to be analyzed for volatile organics constituents (VOCs). VOC analysis requires the collection of one representative grab sample from each source. For new monitoring well installations, the composite IDW sample will include aliquots only from drums associated with that well; however, aliquots from other composite samples may be grouped by AOC or field task. The list of analytes for the composite sample will match that of the primary investigation samples. If significant impacts are encountered during the field investigation, the IDW generated from these sampling locations will be stored in separate containers.

5.1 Liquid IDW Drum Composite Sampling Procedures

The procedures for collecting a sample from liquid IDW drums, with a known source are listed below:

1. Conduct field screening near drum storage area. If elevated concentrations are detected, then increase PPE level to C or B based on the APP.
2. Wearing clean nitrile gloves, remove bung or drum lid and store on plastic sheeting.
3. Dip sample collector/bailer into center of drum and lower the device into the middle section of the drum.
4. Slowly raise the sampling device and decant the appropriate volume into the bottleware.
5. Repeat Steps 3 and 4 until the correct sample volume has been collected. Cap the bottleware between sampling containers.
6. Replace bung or drum lid.
7. Dispose of, or decontaminate, the sampling device.

5.2 Solid IDW Drum Composite Sampling Procedures

The procedures for collecting a sample from solid IDW drums, with a known source are listed below: Conduct field screening near drum storage area. If elevated concentrations are detected, then increase PPE level to C or B based on the APP.

1. Wearing clean nitrile gloves, remove bung or drum lid and store on plastic sheeting.
2. Using a decontaminated trowel, gently scrape the top portion of the drum contents to one side.
3. Place sample collector into center of drum contents and slowly advance the device into the middle section of the drum to a depth of approximately four inches below the surface.
4. Extract the sampling device and transfer the soil to the sample jar.
5. Repeat Steps 4 and 5 until the correct sample volume has been collected. Cap the bottleware between sampling containers.
6. Replace bung or drum lid.
7. Decontaminate sampling device and dispose of plastic as solid PPE, as needed.

Procedures for appropriate sampling nomenclature are presented in Bluestone’s SOP Number 06 – *Field Documentation*.

6.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP Number 05 – *Decontamination of Field Equipment*.

7.0 DATA MANAGEMENT

Pertinent information obtained during IDW characterization sampling and handling included, but not limited to, sample IDs, sample collection times, methods of collection, and PID screening results will be record in the field logbook and on field data sheets as necessary. Bluestone's SOP Number 06 – *Field Documentation* details the methods for data collection and management, and additional information will be specified in the Work Plan. Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photographs must be in accordance with contract requirements and site-specific security requirements.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs.

Standard Operating Procedure Number 12
LOW-FLOW GROUNDWATER SAMPLING AND SAMPLING WITH A BAILER

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the procedures for the collection of groundwater samples using low-flow sampling techniques in both permanent and temporary groundwater monitoring wells. This procedure is consistent with the USEPA SOP: Low Flow (Minimal Drawdown) Groundwater Sampling Procedures (USEPA, 2017). Groundwater samples will be collected from monitoring wells using stainless-steel or polyvinyl chloride (PVC) bladder pumps equipped with Teflon bladders and either polyethylene or Teflon®-lined polyethylene high density tubing. Bladder pumps allow groundwater samples to be retrieved with little disturbance to the sample matrix and minimal exposure to the atmosphere, while employing low-flow techniques.

Groundwater and quality control (QC) samples will be collected and containerized in the order of the volatilization sensitivity of each constituent. If insufficient volume is available to collect the full analytical suite and designated QC samples, the available volume will be allocated at the discretion of the Project Manager and Project Chemist. This hierarchy of samples necessary for low yield/recharged wells will consider factors including, but not limited to, site-specific priorities, sample size, and effect of turbidity on analytical results.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with low-flow or bailer groundwater sampling include biological hazards (poison ivy, bees/wasps, spiders, ticks, etc.) associated with access the well, twisting and repetitive motion (bailing), heaving lifting, nicks and cuts, uneven ground, acid preservatives, contact with contaminated water, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with groundwater sampling, safety glasses, safety boots with ankle support, leather and/or nitrile gloves, and high-visibility vests. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PRE-SAMPLING ACTIVITIES

4.1 Depth-to-Water and Total Well Depth Measurements

Prior to the start of sampling, a synoptic round of depth-to-water measurements will be collected to evaluate the potentiometric surface of groundwater at the site. An electric water-level indicator with an accuracy of +0.01 feet will be used to collect measurements. In addition, the total depth of each well will be measured, and described as a 'hard' or 'soft' bottom to indicate the presence of sediment that may have accumulated at the bottom of the well.

The procedures for collecting depth-to-water measurements include the following:

1. Measurements will be recorded in field logbooks. Depth-to-water and total depth measurements will be collected using the following procedures: Decontaminate the water level probe.
2. Check for proper instrument response by inserting the probe in cup of water. Replace the batteries, as needed.
3. Document observations with respect to the condition of the well pad, surface or protective casing, well locks, obstructions inside the well casing, total well depth, and other well conditions in the field logbook, or on a well assessment field form. Recommendations for maintenance actions should be reported.
4. Don a pair of clean nitrile gloves.
5. Unlock the well cover and remove the cap.
6. Locate the reference point on the riser pipe (notch, mark, or highest point on the riser pipe).
7. Slowly lower the probe down the well until the signal indicates that the water has been contacted.
8. Record the reading at the reference point as depth-to-water.
9. Withdraw the probe and repeat steps 7 & 8. Duplicate measurements should agree within 0.02 feet. If not, continue with measurements until 0.02 feet precision is achieved.
10. Turn the meter to the off position and lower the probe gently until the bottom of the well is encountered. Record the reading at the reference point as the total well depth and observe and record the condition of the bottom of the well (i.e., hard or soft).
11. Remove the probe from the well and decontaminate the unit.

4.2 Field Measurements for Groundwater Sampling

Immediately after removing the well cap, collect a headspace reading using a PID from the top 6 inches inside the well casing. This information will be utilized in conjunction with the site health and safety plan to determine the personal safety level required during sampling. The headspace readings must be recorded in the field logbook. If readings exceed background (0.0 parts per million [ppm]), air monitoring must continue for the duration of the sampling event. Once the headspace reading is recorded, the pump array may be set up and the pump lowered into the well at the target depth.

During the well purging process, water will pass through the flow-through cell at a controlled low rate, and water quality parameters (i.e., temperature, pH, specific conductance, dissolved oxygen (DO), oxidation-reduction potential (ORP), and turbidity) must be recorded every five minutes. The flow-through cell chamber and sample tubing should remain free of air bubbles during the purge process. Tighten or replace the sampling elbow if air bubbles are introduced.

5.0 LOW-FLOW SAMPLING EQUIPMENT

A list of equipment needed for the collection field measurements is summarized below:

- PID
- Multi-parameter water meter (i.e., HORIBA, YSI, or similar) capable of measuring pH, specific conductance, turbidity, temperature, DO, and ORP
- Flow-through cell
- Graduated cylinder
- High-density polyethylene tubing
- 5-gallon pails
- Nitrile gloves
- Pump control box
- Compressed air source

6.0 CALIBRATION OF MULTI-PARAMETER METER

The multi-parameter water meter (HORIBA, YSI or similar) and PID unit must be calibrated at the start of each workday using the factory calibration standards provided by the manufacturer. In addition, ORP will be checked for accuracy using standard redox solution (200-275 millivolts [mV] at 25 degrees Celsius [°C]). Note: Standard Hydrogen Electrode (SHE) corrections for ORP field measurements will be completed at the end of the sampling event. Calibration checks will be performed any time the readings appear erroneous.

DO measurements will be verified both before and after each well. An accuracy check will be performed on the ferrous iron colorimeter once per day. The manual will be consulted if the reading is not within ±0.25 milligrams per liter (mg/L) of the standard solution.

All calibrations and calibration checks will be recorded in the field logbook and appropriate field forms. Extreme cold or hot weather may affect the performance of the YSI meter. In these conditions, the meter may require more frequent checks for instrument drift. Equipment manuals will be available for each instrument, and field personnel should consult the manual for additional technical details. Additional information regarding calibration procedures is available in Bluestone's SOP Number 02 – *Equipment Calibration and Maintenance*.

7.0 LOW-FLOW SAMPLING TECHNIQUES

7.1 Bladder Pump Sampling

A freshly developed monitoring well must be allowed to stabilize for a minimum of 14 days prior to purging and sampling. To the extent possible, monitoring wells will be purged and sampled using bladder pumps. Non-dedicated bladder pumps will be decontaminated prior to and after each use. For bladder pumps used to collect volatile organic constituents (VOCs) or dissolved gas samples, the pump should be set to deliver Begin the task by removing the dedicated pump (if applicable) and collecting the depth-to-water and total well depth measurements, as described above. The pump should not be removed from the well except during the initial round of measurements. Leave the water level indicator in the well and connect the pump and hose assembly to a pump control box. Connect the controller to a compressed gas cylinder containing either nitrogen or carbon dioxide or to a portable air compressor. Connect the flow-through cell to the pump discharge tube so that the sample flows into the bottom of the flow-through cell. Direct the discharge from the flow-through cell into a 5-gallon pail. Groundwater purging will be conducted in accordance with applicable State and/or Federal regulations, as indicated below.

If the project objectives require metal concentrations to be field-filtered, an in-line filter (transparent housing preferred) will be used, in addition to the same low flow procedures. Pre-rinse the filter (0.45 µm) and verify that the filter is free of air bubbles prior to sample collection.

7.2 Procedures for a Sustainable Recovery Well

For the purpose of this SOP, a sustainable recovery well is defined as a well capable of maintaining a stable water level during pumping at a constant flow rate, at an elevation above the pump intake such that there is sufficient volume for all required samples (including any extra volume required for quality assurance/quality control (QA/QC) purposes), plus two additional sampling system volumes. Excessive drawdown refers to drawdown of the water column at a constant flow rate, such that a stabilized water level cannot be obtained at an elevation above the pump intake.

7.3 Standard Sustainable Recovery Well

For this SOP, a standard sustainable recovery well is defined as a well in which stabilized water levels can be obtained at a pumping rate equal to or greater than 100 milliliters per minute (mL/min). The following procedures are used for low flow sampling:

1. Obtain well casing and borehole diameters, and filter pack percent-porosity from available well construction records (may be needed for calculations if the well is determined to be a low- recovery well).
2. Calibrate any electronic water-quality equipment in accordance with manufacturer's instructions and record calibration data in the field logbook and on the calibration form .
3. Check the function of the electronic water level meter in accordance with the

manufacturer's instructions.

4. Assemble equipment at the well and perform field preparatory activities.
5. Measure the total well depth. Using the electronic water level meter, measure and document the depth of the well [to the nearest 0.01 foot (ft)] from the reference mark on the top of the inner well casing.
6. Pump type specific steps.
   a. If using a non-dedicated pump, the following steps are applicable:
      i. Measure water level. Using the electronic water level meter, measure and document the water level to the nearest 0.01 ft. from the reference mark on the top of the inner well casing.
      ii. Assemble the pump and sampling line components, taking care not to contact any of the components with potentially-contaminated media, and ensure that the discharge line is affixed such that initial discharge is captured in either a graduated 5-gallon pail or a purge water collection/disposal drum.
      iii. Determine the depth of the portable pump intake. Measure length of pump from intake to tubing and cable attachment. Measure length of tubing and cable needed to set pump at target depth within the screened interval.
      iv. Slowly lower the pump into the well casing to the target depth, taking care not to encounter the bottom of the well and cause unnecessary agitation of sediment. Affix the pump in this position by fastening the supporting cable. Record depth of pump intake from the reference mark on the top of the inner well casing.
   b. If using a dedicated pump, the following steps are applicable:
      i. Obtain well depth and depth of pump intake from well construction records.
      ii. Measure water level with pump in place. Using the electronic water level meter, measure and document the depth to water (to the nearest 0.01 ft) from the reference mark on the top of the inner well casing.
7. Determine the saturated casing volume and saturated borehole volume (saturated casing volume + saturated filter pack volume). This may be needed for calculations if the well is determined to be a low recovery well.
8. Determine the saturated casing volume above the pump intake. This may be needed for calculations if the well is determined to be a low recovery well.
9. Determine sampling system volume (volume capacity of pump, tubing, and flow-through cell). This may be needed for calculations if the well is determined to be a low recovery well.
10. Determine volume necessary to collect all required samples, including QA/QC samples. This may be needed for calculations if the well is determined to be a low recovery well.
11. Connect the flow-through cell and multi-meter to the pump tubing.

12. If the sustainable flow rate is not known for the well, begin purging at 100 mL/min. For wells with historical sustainable flow rate data, use the historical rate.
13. Ensure that no air bubbles are entrained in the pump tubing. Raise the level of the flow-through cell above the well such that water must pump upward through the intake tubing of the cell. This will purge any bubbles through the tubing. After the cell fills with water, it may be lowered.
14. Measure and record the water level and an initial set of water quality parameter measurements.
15. Determine the initial purge flow rate from the well. Using a graduated cylinder, bucket, or other suitable container of known volume and a stopwatch, time the rate of filling.
16. Determine whether the initial purge flow rate causes excessive water level drawdown in the well. Measure and record the water level and water quality parameters at 500 mL or five-minute intervals . The water level will be considered stable if water level readings do not decrease more than 0.3 ft over three successive measurements (it is acceptable for the water level to remain unchanged or to increase) and if the volume of water in the casing above the pump intake is equal to or greater than the volume needed for all required samples plus two sampling system volumes.
17. If the initial purge rate of 100 mL/min does not cause excessive drawdown and is an appropriate rate for project analytes and purposes, document that sustainable recovery has been achieved at this rate and skip to Number 21 and obtain stabilized water quality parameter readings.
18. If the initial purge rate of 100 mL/min does not cause excessive drawdown and a higher rate is desirable for project-specific reasons, adjust the flow rate and determine whether sustainable recovery can be obtained using the higher flow rate. Record each adjustment made to the pumping rate, the water level, and the multi-meter readings measured immediately after each adjustment. The water level and water quality parameters should be measured and recorded approximately every five minutes. When sustainable recovery has been documented at the higher flow rate, skip to Number 21 and obtain stabilized water quality parameter readings. (Note: Assuming a highly transmissive formation, one liter/minute is the maximum purge rate that will preserve laminar flow in the screened interval).

If the initial purge rate of 100 mL/min causes excessive drawdown and the well is less than 30 feet deep, the procedure may be repeated using a peristaltic pump to determine whether sustainable recovery can be obtained at flow rates less than 100 mL/min (See alternative sustainable recovery well section below).

19. If the initial purge rate of 100 mL/min causes excessive drawdown and alternative equipment with flow rates less than 100 mL/min cannot be used, see Low-Recovery Wells Section below.
20. Once a stabilized water level has been obtained, the field parameters will be monitored for stabilization. If the flow rate is equal to or greater than 100 mL/min, measure and record the water quality parameters at five-minute intervals. If the flow rate is less than

100 mL/min, record the water quality parameters at time intervals of 500 mL divided by the purge rate. Field parameters will be considered stable when three consecutive measurements within the following ranges are obtained:

a. Turbidity: (10% for values greater than 5 Nephelometric turbidity units (NTU), if three turbidity values are less than 5 NTU, consider the values as stabilized),
b. Dissolved Oxygen: (10% for values greater than 0.5 mg/L, if three DO values are less than 0.5 mg/L, consider the values as stabilized),
c. Oxidation/Reduction Potential: (+/- 10 millivolts),
d. Specific Conductance: (3%),
e. pH: (+/- 0.1 unit), and
f. Temperature: (3%).

21. Once water quality parameters have stabilized, the groundwater sample may be collected.

If parameters other than turbidity stabilize, but turbidity stabilization cannot be attained, the Project Manager will be consulted.

7.4 Alternative Sustainable Recovery Well

An alternative sustainable recovery well is defined as a well in which stabilized water levels can be obtained at a pumping rate less than 100 mL/min using alternative equipment capable of lower flow rates (e.g., peristaltic pump, mini bladder pump). The following procedures will be used for alternative sustainable recovery wells:

1. If stabilized water levels can be obtained at a pumping rate less than 100 mL/min using alternative equipment, refer to Number 21 above, and obtain stabilized water quality parameters. Note that for flow rates of less than 100 mL/min, parameter measurement interval is determined by 500 mL divided by the purge rate.

7.5 Procedures for Sampling Low-Recovery Wells

A low recovery well is defined as a well in which stabilized water levels cannot be obtained as described for sustainable wells, regardless of pumping rate or equipment type. The following procedures will be used for low recovery wells:

1. If a purge rate of 100 mL/min causes excessive drawdown and/or alternative equipment with flow rates less than 100 mL/min cannot be used, the following procedures should be used.
2. The following information (see previous steps 1, 7, 8, 9) is needed:
   - Obtain well casing and borehole diameters, and filter pack percent-porosity from well construction records.
   - Determine saturated casing volume and saturated borehole volume (casing volume + saturated filter pack volume).
   - Determine saturated casing volume above the pump intake.
   - Determine sampling system volume (volume capacity of pump, tubing, and flow-through cell).

- Determine volume necessary to collect all required samples, including QA/QC samples.
- Determine whether the saturated casing volume above the pump intake is sufficient for at least two sampling system volumes plus required samples.

3. If the casing volume above the pump intake is sufficient for at least two sampling system volumes plus required samples, purge slowly at a constant flow rate. measure and record water levels and field parameters every 500 mL until two (or available) system volumes have been removed; collect samples; document conditions and procedures. (Note: water level will not be stable (i.e., drawdown will occur) and water quality parameters may not be stable).
4. If the casing volume above the pump intake is not sufficient for at least two sampling system volumes plus required samples but is sufficient for at least one sampling system volumes plus required samples, purge slowly at a constant flow rate. measure and record water levels and water quality parameters every 500 mL until one (or available) system volumes have been removed; collect samples; document conditions and procedures. (Note: water level will not be stable (i.e., drawdown will occur) and water quality parameters may not be stable).
5. If the casing volume above the pump intake is sufficient for required samples only, determine whether it is acceptable to collect samples without purging. If this is acceptable for project purposes, collect samples at a constant flow rate without purging, document conditions and procedures.
6. If the casing volume above the pump intake is not sufficient for required samples, determine whether samples can be prioritized and if it is acceptable to collect priority samples without purging. If this is acceptable for project purposes, collect the priority samples at a constant flow rate without purging, document conditions and procedures.
7. If the casing volume above the pump intake is not sufficient for all required samples, samples cannot be prioritized, and/or it is not acceptable for project purposes to collect samples without purging, do not collect a sample, and document conditions.
8. If the well cannot be sampled using the low-recharge procedure:
    - Determine whether diffusion samplers or other passive methods are acceptable for project purposes and can be used.
    - Determine whether the well can be removed from the monitoring network.

8.0 GROUNDWATER SAMPLING DECISION TREES

The following decision tree will be used for purging monitoring wells (see Figures 1 and 2 below):

1. Wells with historical purge rate data:
    - For a consistent, sustainable recovery well (i.e., stabilized water level can always be achieved) using either standard or alternative equipment.
        - Use historical sustainable flow rate and equipment
        - Obtain stabilized water level

        - Obtain stabilized water quality parameters; and
        - Collect samples.
    - For a well with inconsistent recovery across multiple sampling events, determine whether a sustainable flow rate can be achieved during this sampling episode using standard or alternative equipment.
        - If yes, use sustainable rate and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedure.

2. Wells without historical purge rate data but with information from well development or redevelopment:
    - Does well development or redevelopment record indicate a sustainable recovery well using either standard or alternative equipment?
        - If yes, determine a sustainable flow rate and use sustainable rate procedures and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedures.
3. Wells without either historical purge rate or well development or redevelopment data:
    - Determine whether a sustainable flow rate can be achieved during this sampling event using standard or alternative equipment.
        - If yes, use the sustainable rate and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedures.

9.0 EQUIPMENT MALFUNCTION PROCEDURES

Every effort will be made to procure and maintain properly functioning equipment; however, equipment malfunctions may occur. In these instances, the field team leader will be contacted immediately, followed by the Project Manager. To the extent practicable, field crews will be equipped with backup sets of equipment. Any necessary replacement items will be ordered for next-day delivery. Since the measurement of DO is the most frequent failure point, subsequent wells will be purged and sampled at or below historical purge rates until 125 percent of the maximum volume purged during the previous three sampling events is removed from the well. All other stabilization parameters must also meet stabilization criteria prior to sample collection.

Any other equipment malfunctions will be brought to the attention of the Project Manager and a temporary site-specific sampling protocol will be implemented. In addition, any equipment malfunctions and remedies must be noted in the field logbook and on the daily report.

Figure 1 Groundwater Sampling Decision Flowchart - Wells Screened Below Water Table

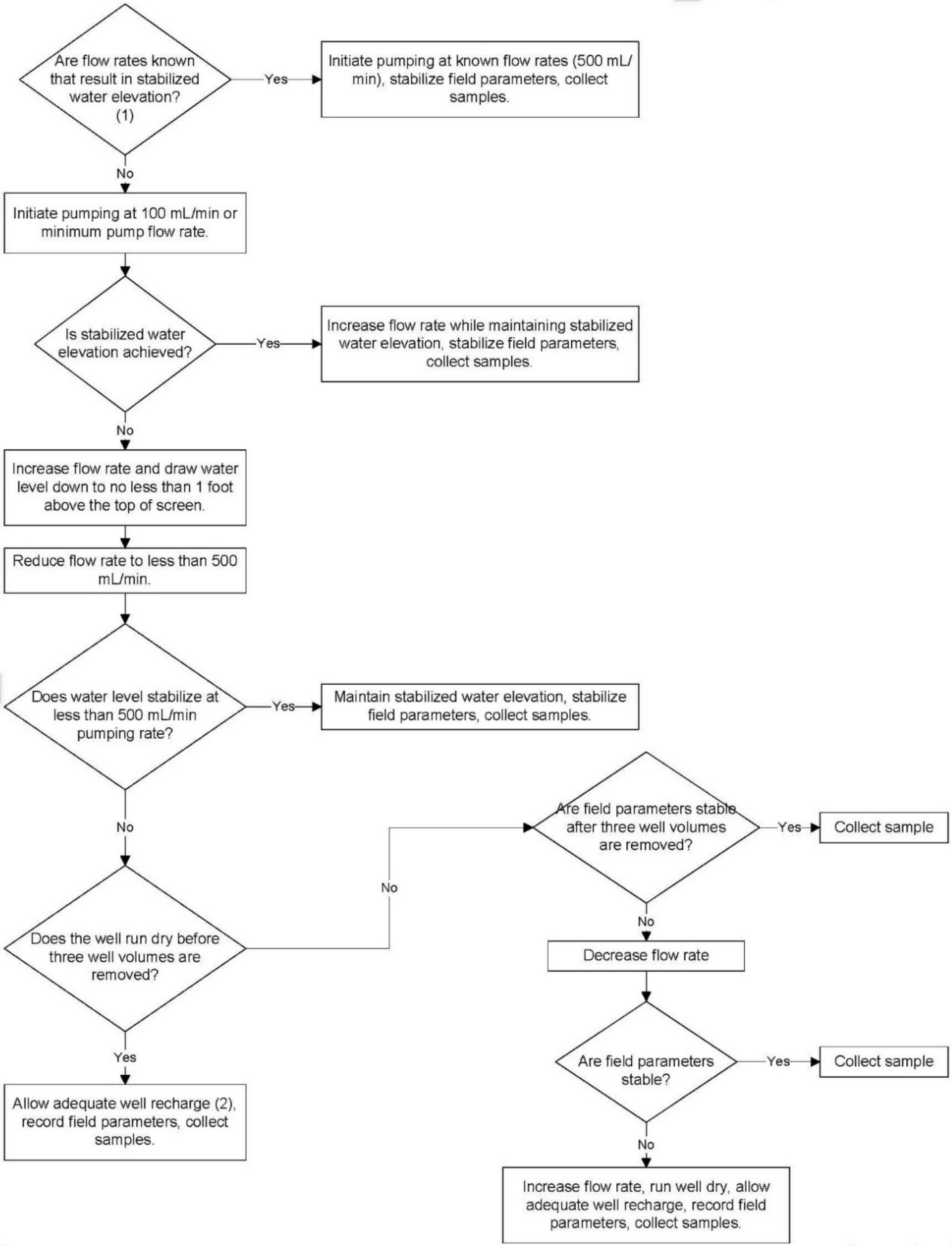
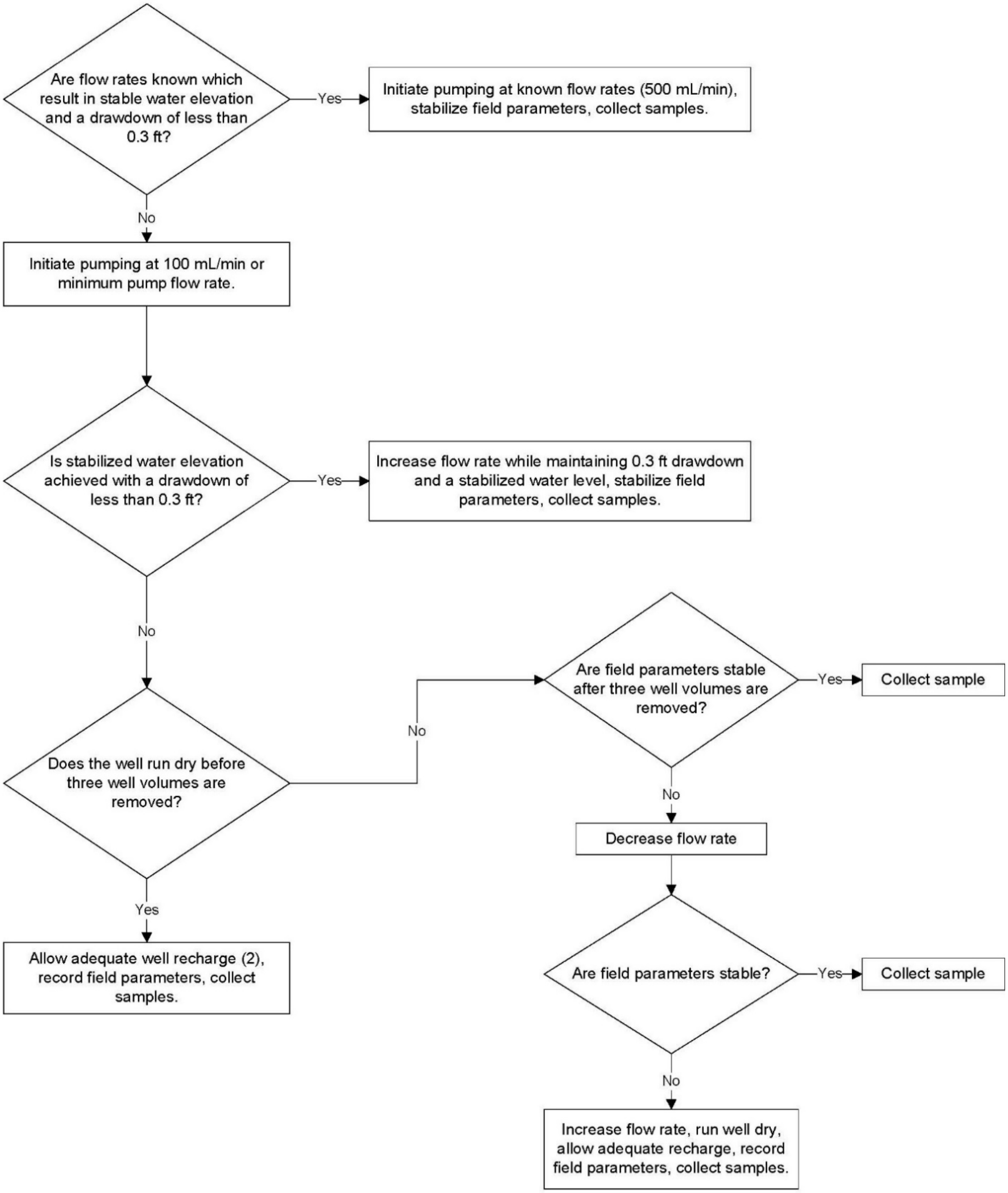


Figure 2 Groundwater Sampling Decision Flowchart - Wells Screened Across Water Table



10.0 GROUNDWATER SAMPLE COLLECTION USING A BAILER

For groundwater samples collected with a bailer, a standard cleaned closed-top polyethylene or Teflon® bailer with Teflon® coated stainless-steel leaders must be used. A new piece of nylon rope must be used for each bailer. The bailer and rope are lowered slowly into the well to the top of the water column, allowed to fill, and removed. It is critical that the bailer be slowly and gently lowered into the water column, particularly during the final stages of purging, to minimize turbidity and disturbance of any VOCs. A straight tube should be used to displace the check valve at the bottom of the bailer to decant the water into sample the appropriate sample containers.

11.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP Number 05 – Decontamination of Field Equipment.

12.0 DATA MANAGEMENT

Data collected during well sampling activities (i.e., depth-to-groundwater measurements, total well depth, sampling IDs, sample collection times, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP Number 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

13.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

Standard Operating Procedure Number 13
SAMPLE PACKAGING AND SHIPPING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the procedures for the proper packaging and shipping of environmental samples. All samples must be shipped priority overnight in accordance with the U.S. Environmental Protection Agency (USEPA) specifications and U.S. Department of Transportation (DOT) regulations (49 Code of Federal Regulations [CFR] Parts 172 and 173). Samples will be handled as a low hazard level, and packed and shipped within 24 hours of collection.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SSHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with packing and shipping samples include primarily nicks and cuts, and heavy lifting. Modified Level D is generally appropriate, and may be limited to nitrile gloves, safety glasses and safety shoes. High visibility vests or clothing will be worn when packing coolers in a parking lot. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 SAMPLE PACKAGING

The following procedure will be used to pack samples for overnight shipment:

1. To the extent possible, group samples by sample ID, with the exception of bottleware designated for volatile organic constituents (VOC) analysis. Group aqueous VOC samples in a common shipping container.
2. At the time of sample collection, wipe the outside of each sample bottle/jar with a paper towel and place a label on each container. Each glass bottle/jar will be wrapped with bubble wrap or the protective sleeves provided by the laboratory. Place all sample bottles in a Ziploc® bag. All VOC vials collected for each sample will be placed in the same plastic bag, if practicable. Trip blank quality control (QC) samples will be packed in the same manner as the VOC bottleware.

3. Remove as much air as possible from the plastic bag prior to sealing.
4. Seal the drains on the shipping coolers with tape.
5. Place an absorbent pad in the bottom of the cooler, followed by a layer of bubble wrap.
6. Insert a plastic liner/contractor bag into the cooler.
7. Place the sample containers inside the liner in an upright position and place sections of bubble wrap in between sample bottles. Group all aqueous VOC samples in a common cooler. Place one trip blank set (two 40-mL volatile organic analysis [VOAs]) in each cooler containing aqueous VOC samples. Place one temperature blank in each cooler.
8. Preserve the samples with ice,. This may be completed by placing the ice chips directly inside the liner around all sample bottles or by double-bagging ice chips in Ziploc® bags and placing the bags at the bottom, top, and in the interstitial spaces of sample batch.
9. Sign and date the chain of custody (COC) and record the information in the field logbook.
10. If a laboratory courier service is arranged, the sampler and courier must both sign and date the COC at the time of transfer. The sampler must then take a photo of the COC before sealing it inside the cooler. Photographs of COCs will be provided to the sample manager at the end of each workday.
11. To properly seal the completed COC inside a cooler, separate the bottom copy and place the remaining pages inside a Ziploc® bag and tape the bag to the inside of the cooler lid.
12. Affix signed custody seals over lid openings (opposite corners of the cooler).
13. Seal both ends of the cooler by wrapping three times with clear packing tape. Make sure that the address, phone number, and contact information for the receiving laboratory is placed prominently on the cooler, and wrap shipping label with packing tape.
14. For shipments through FedEx or UPS, provide the representative with the laboratory shipping and receiving addresses and the Bluestone or laboratory account number, depending on responsible party. In addition, provide any shipping procedures or restrictions the laboratory may require (i.e. no Saturday delivery).

5.0 DATA MANAGEMENT

Data associated with the release of samples to a courier service will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP Number 06 – Field Documentation details the methods for data collection and management, and additional information should be specified in the Work Plan.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

Standard Operating Procedure Number 19
GROUNDWATER ELEVATION MONITORING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for collecting depth-to-water measurements from monitoring wells and piezometers (terms used interchangeably). Hydrogeologic data gathered during a field investigation may be used to determine hydraulic gradient, interpret the migration rate and direction of contaminants (if present), produce groundwater contour maps, determine purge volumes for groundwater sampling, and design slug tests, packer tests and constant-rate pumping tests

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, the site safety and health officer (SHHO) must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Site Safety and Health Plan (SSHP), the Bluestone Corporate Safety and Health Program, and site and/or client-specific requirements. Potential health and safety hazards associated with groundwater elevation monitoring include biological hazards including, but not limited to, poison ivy, stinging insects, spiders, and ticks; slip/trip/fall hazards; and pinch points. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with groundwater elevation monitoring, and includes a hard hat, safety glasses, leather/nitrile gloves, safety shoes and high-visibility vests.

4.0 EQUIPMENT

Equipment used in the collection of depth-to-water measurements:

- Electronic water level indicator (Solinst® Model 101 P7 Water Level Meter or similar)
- Oil/water interface probe (Solinst® Model 122 or similar)
- Alconox®, Liquinox® or other non-phosphate concentrated laboratory grade soap
- De-ionized water
- Spray bottles
- PPE
- Air Monitoring instruments as required (i.e. MiniRAE®, Thermo TVA 1000 FID/PID)
- Field logbook / field data sheets
- Well keys
- Decontamination supplies (SOP Number 05 – *Decontamination of Field Equipment)*
- Previous depth-to-water measurements (if available).

5.0 PROCEDURES FOR MEASURING DEPTH-TO-WATER

The procedures for collecting depth-to-water measurements include the following:

1. Record the condition of the well (protective casing, concrete collar, lock in place, etc.).
2. Check the water level tape has no obvious kinks or damage.
3. Wearing nitrile gloves, decontaminate the water level meter in accordance with Bluestone's SOP Number 05 – *Decontamination of Field Equipment*.
4. Stand upwind of the well; unlock and open the well, being cautious of biological hazards. Remove the well cap and collect a headspace reading using a PID. Record pertinent air monitoring data (sustained, dissipating, background, odor) in the field logbook in accordance with Bluestone's SOP Number 06 – *Field Documentation*.
5. Identify the survey notch on the riser or casing (if present). Record this location in the field logbook or field data sheet.
6. Activate the water level meter, check the audible indicator, reel the electronic probe into the well riser (with the increments visible) slowly until the meter sounds, grasp the tape with hand, withdraw the tape and lower it again slowly until the sound is again audible. Check the depth to water on the tape and make a mental note of the depth to within .01 feet. Lower the probe again slowly and repeat the measurement for accuracy, with care to measure from the correct direction.
7. Record the depth-to-water measurements in the field logbook or field data sheets and corresponding time of measurement.
8. Procedures implemented in the presence of free phase petroleum products (light non-aqueous phase liquids [LNAPL]) on the surface of the water table will be modified to include the use of the oil/water interface probe. The procedures during the use of this probe will be implemented similarly and by manufacturers' specifications. Typically, the procedure to collect depth-to-product measurements is similar to measuring depth-to-water, where the oil/water interface probe will be equipped with a dual alarm that distinguishes between contact with oil and contact with water. Depth-to-oil and depth-to-water measurements will be recorded, which in turn provides the thickness of the free product layer.

6.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP Number 05 – *Decontamination of Field Equipment*.

7.0 DATA MANAGEMENT

Data collected during groundwater elevation monitoring (i.e., depth-to-groundwater and total depth measurements, and corresponding times, etc.) will be recorded in the field logbook and on field data sheets, as necessary. Bluestone's SOP Number 06 – *Field Documentation* details the methods for data collection and management, and additional information will be specified in the Work Plan. Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photographs must be in accordance with contract requirements and site-specific security requirements.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs.

Appendix B
Laboratory SOPs

Title: ACID DIGESTION OF AQUEOUS SAMPLES

Method(s): SW846 3005A and 3010A and EPA Methods 200.7 and 200.8

Approvals (Signature/Date):

	11/11/2019		11/6/2019
Roseann Ruyechan Inorganics Department Manager	Date	Steve Jackson Regional Safety Coordinator	Date
	11/4/2019		12/3/2019
Virginia Zusman Quality Assurance Manager	Date	Deborah L. Lowe Laboratory Director	Date

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. d/b/a Eurofins TestAmerica and its affiliates (“Eurofins TestAmerica”), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Eurofins TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically provided. The user also agrees not to give access to this document to any third parties including but not limited to consultants, unless such third parties specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2019 TESTAMERICA LABORATORIES, INC. d/b/a EUROFINS TESTAMERICA ALL RIGHTS RESERVED.

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and ICPMS using EPA Methods 200.7, 200.8 and SW846 Methods 3005A and 3010A.

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II in Appendix A. Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, total recoverable and total metals elements in surface water, ground water, aqueous samples, leachates/extracts.

1.4. SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP or ICPMS.

1.5. ICP Method 200.7 and ICPMS Method 200.8 are used to prepare surface water, domestic and industrial waste samples for total recoverable and dissolved metals.

1.6. SW-846 Method 3010A is used to prepare TCLP Leachates for total metals analysis by ICP or ICPMS.

1.7. When dissolved metals analysis is requested the samples must be field filtered at the time of collection and prior to preservation.

1.8. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.9. Quantitation limits can be found in the analytical method SOPs, PT-MT-001 and PT-MT-002.

1.10. On occasion clients may request modifications to this SOP. Any modifications to this procedure must be approved by the QA department of the Laboratory or Technical Director, and be documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. Method 3005A for Method 6010B/C ICP or 6020/6020A ICPMS - Preparation for Total Recoverable or Dissolved Metals Analysis

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A - Preparation for Total and TCLP Metals Analysis by Method 6010B/C/D ICP or 6020/6020A/B ICPMS

2.2.1. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to volume.

2.3. Methods 200.7 and 200.8 have method specific preparations included.

2.3.1. A representative samples, with <1% suspended solids, is refluxed with nitric and hydrochloric acid and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.4. Refer to PT-QA-024 for subsampling procedures.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements that pass through a 0.45 um membrane. (Sample is acidified AFTER filtration by filtering into a nitric acid preserved bottle).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.5. TALS: TestAmerica Laboratory Information Management System

3.6. NCM: Non-Conformance Memo, an electronic memo from the lab to reviewers and Project Managers, to notify them of a non-conformance, anomaly, or special condition associated with a sample or analytical batch.

3.7. Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M- 001) current version, for additional definitions.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Glass and metal labware and powdered gloves are avoided in this area to reduce contamination chances.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix C for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they are documented in a sample NCM. Reactions or anomalies such as foaming, emulsions, precipitates, etc., must also be documented.

4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.6. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. If this is suspected, samples may be re-digested at a dilution.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-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.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor and the EHSC.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Hot block, or other adjustable heating source capable of maintaining a temperature of 80 to 95°C

6.2. Thermometer that covers a temperature range of 0-150°C

6.3. Hot Block Disposable Digestion Tubes (from Environmental Express), 50mL, with nominal volume marking certified within Class A tolerance

6.4. Watch glasses, plastic disposable (from Environmental Express)

6.5. Plunger filters, plastic disposable (from Environmental Express)

6.6. Fisherbrand Q8 filter paper, or Whatman No. 41, or equivalent

6.7. Funnels or equivalent plastic filtration apparatus

6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.9. Re-pippetors or suitable reagent dispensers

6.10. Calibrated automatic pipettes with corresponding pipet tips

6.11. Class A volumetric flasks

6.12. pH indicator strips (pH range 0 - 14)

6.13. Plastic digestate storage bottles (from Environmental Express)

7. REAGENTS AND STANDARD

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

All standards and reagents for this procedure are stored at room temperature, unless otherwise specified by manufacturer requirements.

7.1. Reagent water must be produced by a Millipore type deionization system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom stock standard mix solutions. All standards must be stored in the original container or in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from receipt and must be replaced sooner if verification from an independent source indicates a problem.

7.2.1. Working ICP/ICPMS LCS/MS spike solution: The LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.2.2 The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom TestAmerica solution, a solution must be purchased from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.2.3 Aqueous laboratory control samples and matrix spike samples are prepared as described in Sections 9.4 and 9.5. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP/ICPMS LCS and matrix spike preparations.

7.3. The TCLP MS spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. Nitric acid (HNO_3), concentrated, trace metal grade and Ultrapure

7.4.1. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.5. Hydrochloric acid (HCl), concentrated, trace metal grade and ultra-pure (Omnitrace)

7.6. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.7. All applicable acids and reagents used for ICPMS digestion are ultra-pure reagents.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Water	250 mL Plastic bottle	100 mL	pH <2 preserved with HNO_3	180 days from collection to analysis	40 CFR Part 136

8.1. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified at the time of receipt by the laboratory.

8.1.1. If the pH of the aqueous is found to be > 2 at the time of receipt, Sample Receiving will add preservative, document the amount of acid added and the time it was added, See Figure 5. The sample will be held for 24 hours from the documented preservation time before proceeding with the sample preparation.

8.2. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration should be done in the field within 15 minutes of collection. If the laboratory filters the samples, this process will occur as soon as feasibly possible after receipt, and samples must not be preserved until after filtration.

8.2.1. Dissolved metals samples that are filtered directly into a nitric acid preserved bottle may be digested after filtration is complete.

8.2.2. If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

8.2.3. If samples are received unpreserved and are filtered in the lab for dissolved metals, include the "Filtration – Field Filtration, 15 Minutes" NCM on the filtration batch.

9. QUALITY CONTROL

Summary of Batch QC

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA(1)	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PT-MT-001 and PT-MT-002	Redigest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs:	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples (SW-846 Methods) or one per every 10 or fewer samples (200.7/200.8).	Refer to determinative SOPs:	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs:	See Corrective Action for Matrix Spike.

9.1. Preparation Batch - A group of up to 20 samples that is of the same matrix and is processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate (SW-846 Methods) or a matrix spike for every 10 or fewer sample (200.7/200.8). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. The same lot of reagents (acids) must be used to prepare batch QC and all samples in the batch.

9.2. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS, MSD) are not included in the sample count for determining the size of a preparation batch.

9.3. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire preparation and analytical procedure. The method blank is used to identify any system and process interferences or contamination that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.3.1. Aqueous method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 10.

9.3.2. TCLP method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 10. The TCLP Leachate Blank is prepared in the same manner as the leachate samples.

9.4. Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in methods 200.7/200.8) must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire preparation and analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be re-preparation and reanalysis of the batch. Refer to Section 7.2 for instructions on preparation of the aqueous LCS spike solution.

9.4.1. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water or TCLP leachate blank with 0.25 mL of the appropriate stock spike mix solution(s) (7.2). The LCS is then processed through the appropriate procedure as described in Section 10.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples (SW-846 Methods) or one matrix spike is processed for every 10 or fewer samples (200.7/200.8). A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 200.7/200.8). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5.1. The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 0.25 mL of the appropriate stock spike mix solution(s) (7.2). The matrix spike sample is then processed as described in Section 10.

9.5.2. The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.25 mL of the TCLP spike solution (7.3). The matrix spike sample is then processed as described in Section 10.
NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.5.3. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Document this situation using the "MS/MSD/DUP-Insufficient Volume" NCM.

10. PROCEDURE

10.1. Hotblock temperature must be verified daily for each hotblock used and must be recorded in the batch information in TALS. The hotblock temperature should be verified by measuring the temperature of a sample container of reagent water placed on each hotblock. The position will be changed daily in order to monitor the temperature of the entire hotblock. This position ID will also be recorded in the TALS batch information and on the Hotblock diagram (see Appendix D).

10.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3. All digestion procedures must be carried out in a properly functioning hood.

10.4. All samples are to be checked out of sample control with the TALS ICOC.

10.5. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample. Do not label digestion tubes below the level where they will be inside the hot block.

10.6. Samples are typically logged in as either waters or wastes. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

10.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab.

10.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

10.9. The following procedure must be followed for all aqueous sample preparations:

10.9.1. Measure and record sample pH with pH paper on a separate aliquot of sample. This is typically verified and documented at sample receipt. If samples were found to not be properly preserved at receipt, and the laboratory was instructed by the client to correct the preservation, the sample is transferred to a fresh nitric acid preserved bottle and marked with a purple dot to indicate it was preserved upon receipt. These samples must not be digested for at least 24 hours after preserving. Samples preserved at receipt have pH rechecked in the lab prior to digestion.

Note: If the sample pH is > 2 pH units, the client must be notified of the anomaly before digestion proceeds.

10.9.2. Mix sample by shaking the container.

10.9.3. Measure and transfer 50 mL of the sample into a digestion tube.

Note: For TCLP samples, a 5 mL sample volume, diluted to 50mL with reagent water, will be digested to minimize matrix interferences for filtered waters and tumbled solids (leachates).

10.9.4. Measure extra aliquots of sample(s) selected for the MS/DUP or MS/MSD analysis. Spike each MS and/or MSD aliquot with 0.25 mL of spiking solution (7.2 or 7.3).

10.9.5. Measure and transfer 50 mL of reagent water into a digestion tube for the method blank.

10.9.6. Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add 0.25 mL of spiking solution (7.2)

10.10. Proceed to the appropriate Section for the desired method as follows:

Method 3005A	10.11
Method 3010A	10.12
Method 200.7	10.13
Method 200.8	10.14

NOTE: When filtering the final digestates from the processes below, if there is significant suspended matter in the digestate, pre-washed (1% nitric acid) Fisherbrand Q8 filter paper in a plastic funnel may be used in place of the Environmental Express plunger filters.

10.11. Method 3005A - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP/ICPMS (See Figure 1)

10.11.1. To 50mL of sample in the digestion tube, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

10.11.2. Cover with disposable watch glass.

10.11.3. Heat at 90 - 95°C in the hotblock until volume is reduced to 10 mL or less. **DO NOT ALLOW SAMPLE TO BOIL OR GO DRY.** Doing so will result in the loss of analyte and the sample must be re-prepared.

10.11.4. Cool the digestion tubes in a fume hood.

10.11.5. Wash down digestion tube walls and watch glass with reagent water.

10.11.6. Adjust the final volume/mass to 50 mL with reagent water. The sample is now ready for analysis

10.11.7. If insoluble materials are present, plunge filter samples after it has been brought to volume. Label the cap of the filtered sample as “PF” to let the instrument analyst know why there is not a full 50mL of digestate.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered. The LCS will have been spiked with the analytes of interest prior to digestion and filtering in order to assess the effectiveness of the entire preparation procedure.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.12. Method 3010A - Preparation for Total and TCLP Metals Analysis by ICP/ICPMS Spectroscopy (See Figure 2)

10.12.1. To 50mL of sample in the digestion tube, add 1.5 mL of concentrated HNO_3.

NOTE: For TCLP samples, a 5 mL sample volume diluted with reagent water will be digested to minimize matrix interferences from wastes and leachates.

10.12.2. Cover with disposable watch glass.

10.12.3. Place digestion tube into the hotblock (90-95°C) and evaporate to 5 - 10 mL while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.12.4. Cool the digestion tubes in a fume hood.

10.12.5. Add another 1.5 mL portion of concentrated HNO_3 and re-cover the digestion tube.

10.12.6. Continue refluxing until the digestion is complete.

Note: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient, additional aliquots of nitric acid may be added if necessary.

10.12.7. Evaporate to low volume of 10 mL or less while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

10.12.8. Cool the digestion tube in a fume hood.

10.12.9. Add 5 mL of 1:1 HCl.

10.12.10. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue.

10.12.11. Wash down digestion tube walls and watch glass with reagent water.

10.12.12. Rinse digestion tube and filter paper or plunger filter with reagent water to ensure complete sample transfer.

10.12.13. Adjust final volume/mass to 50 mL with reagent water. The sample is now ready for analysis.

10.12.14. If insoluble materials are present, plunge filter samples after it has been brought to volume. Label the cap of the filtered sample as “PF” to let the instrument analyst know why there is not a full 50mL of digestate.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered. The LCS will have been spiked with the analytes of interest prior to digestion and filtering in order to assess the effectiveness of the entire preparation procedure.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.13. Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP (See Figure 3)

10.13.1. To a digestion tube containing 50 mL of sample, add 1 mL of 1:1 HNO_3 and 0.5 mL of 1:1 HCl.

10.13.2. Heat at 80-85 °C for uncovered digestion tubes, or 90-95℃ for cov ered digestion tubes, until volume is reduced to about 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

NOTE: If a sample contains > 1% suspended solid material by visual inspection, add a sample volume containing no more than 1.0 grams of solid to the digestion tube. Increase the acid volume to 4.0 mL of 1:1 HNO_3 and 4.0 mL of 1:1 HCl and proceed with digestion.

10.13.3. Cover with disposable watch glass.

10.13.4. Gently reflux for 30 minutes.

10.13.5. Cool the digestion tube in the fume hood.

10.13.6. Wash down digestion tube walls and watch glass with reagent water.

10.13.7. Rinse digestion tube and plunger filter with reagent water to ensure complete sample transfer.

10.13.8. Adjust the final volume/mass to 50 mL with reagent water. The sample is now ready for analysis.

10.13.9. If insoluble materials are present, plunge filter samples after it has been brought to volume. Label the cap of the filtered sample as "PF" to let the instrument analyst know why there is not a full 50mL of digestate.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered. The LCS will have been spiked with the analytes of interest prior to digestion and filtering in order to assess the effectiveness of the entire preparation procedure.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.14. Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICPMS (See Figure 4)

10.14.1. To the digestion tube containing 50 mL of sample, add 1 mL of 1:1 HNO_3 and 0.5 mL of 1:1 HCl.

10.14.2. Heat at 80-85 °C for uncovered digestion tubes or 90-95°C for covered digestion tubes until volume is reduced to about 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

NOTE: If a sample contains > 1% suspended solid material by visual inspection, add a sample volume containing no more than 1.0 gram of solid to the digestion tube. Increase the acid volume to 4.0 mL of 1:1 HNO_3 and 4.0 mL of 1:1 HCl and proceed with digestion.

10.14.3. Cover with disposable watch glass.

10.14.4. Gently reflux for 30 minutes.

10.14.5. Cool the digestion tube in the fume hood.

10.14.6. Wash down digestion tube walls and watch glass with reagent water.

10.14.7. Rinse digestion tube and plunger filter with reagent water to ensure complete sample transfer.

10.14.8. Adjust the final volume/mass to 50 mL with reagent water. The sample is now ready for analysis.

10.14.9. If insoluble materials are present, plunge filter samples after it has been brought to volume. Label the cap of the filtered sample as “PF” to let the instrument analyst know why there is not a full 50mL of digestate.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered. The LCS will have been spiked with the analytes of interest prior to digestion and filtering in order to assess the effectiveness of the entire preparation procedure.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.15. Documentation and Record Management

10.15.1. All information is recorded directly into TALS prep worksheet. Reagent information is documented in the batch information and spiking solutions are documented on the reagent tab in the batch.

11. CALCULATIONS / DATA REDUCTION

11.1. Not Applicable

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. A Method Detection Limit (MDL) study, as described in Eurofins TestAmerica corporate Detection Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change is made to the analytical system. The MDL must be verified at leasat every 12 months.

13. POLLUTION CONTROL

13.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to CW-E-M-001. The following waste streams are produced when this method is carried out.

14.1.1. Acidic waste containing nitric acid generated by the digestion. This waste is collected in a waste container identified as "Acid Waste", Waste #33. This waste is neutralized to a final pH between 5 and 9 and discharged down into a lab sink.

14.1.2. Contaminated disposable materials utilized for the analysis. These items are placed in trash containers which are emptied in the general trash dumpster located near the shipping/receiving dock.

15. REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

15.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Method 200.7, Revision 4.4, May 1994

15.3. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4, 1994

15.4. PT-MT-001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B/6010C/6010D and Method 200.7

15.5. PT-MT-002, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020/6020A/6020B

15.6. PT-QA-021, TestAmerica Pittsburgh QC Program

15.7. CA-Q-S-006, corporate Detection Limits SOP

15.8. PT-QA-024, Subsampling

15.9. PT-QA-M-001, TestAmerica Pittsburgh's Laboratory Quality Assurance Manual

15.10. PT-QA-016, Nonconformance & Corrective Action System

15.11. PT-QA-001, Employee Orientation and Training

15.12. PT-HS-001, Appendix D, Waste Collection, Accumulation and Storage

15.13. PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.14. PT-QA-031, Internal Chain of Custody

16. METHOD MODIFICATIONS

16.1. Modifications applicable to SW-846 reference methods.

16.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.1.2. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "..flexibility to alter digestion volumes is addressed and "allowed" by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Olliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

16.2. Modifications Specific to Method 3010A

16.2.1. Section 10.12.7 of this SOP requires the sample be reduced to a volume of 10 mL or less. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the digestion tube should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the digestion tube so as to prevent the loss of critical analytes through volatilization.

16.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17. ATTACHMENTS

17.1. Figure 1 – Method 3005A Flowchart
17.2. Figure 2 – Method 3010A Flowchart
17.3. Figure 3 – Method 200.7 Flowchart
17.4. Figure 4 – Method 200.8 Flowchart
17.5. Figure 5 – Example of a Lab Aqueous pH Preservation Form
17.6. Appendix A – Tables
  17.6.1. Table I – Approved Preparation Method Analytes – SW846
  17.6.2. Table II – Approved Preparation Method Analytes – NPDES
  17.6.3. Table III – ICP/ICPMS Matrix Spike and Aqueous Laboratory Control Sample Levels
  17.6.4. Table IV – TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
  17.6.5. Table V – Summary of Quality Control Requirements

17.7. Appendix B – TALS Metals Prep Example Worksheets
17.8. Appendix C – Contamination Control Guidelines
17.9. Appendix D – Example Template for Hotblock Thermometer Rotation

18. REVISION HISTORY

18.1. Revision 7, 8/24/07
18.2. Revision 7.1, 10/11/07
18.3. Revision 8, 1/31/2009
18.4. Revision 9, 2/8/2010
18.5. Revision 10, 6/23/2010
18.6. Revision 11, 5/8/2012
18.7. Revision 12, 6/22/2014
18.8. Revision 13, 7/26/2016
18.9. Revision 14, 10/5/2018
18.10. Revision 14.1, 4/25/19
18.11. Changes to current revision

SOP section	**Change from**	**Change to**	**Reason**
1.9		Added reference to quantitation limits in SOPs PT-MT-001 & -002	Clarification
1.10	Modifications per QA Manual	Modifications must be approved and documented	Clarification
2.2		Added 6010D and 6020B	Update
7		Added to store all standards at room temperature unless otherwise specified	Clarification
9.4.1, 9.5.1, 9.5.2, 10.9.4, 10.9.6	0.5 mL spike	0.25 mL spike	New standards
10.11.7, 10.12.17, 10.13.9, 10.14.9		Added if insoluble matter is present, to plunge filter samples after bringing to volume and write "PF" on cap	Correction
10.2	MDL per QAM and SOP PT-QA-007	MDL per corp. SOP CA-Q-S-006	Update
15.7	PT-QA-007	CA-Q-S-006	Update
Table III		Updated all spikes for new standard of standards stocks	Update

FIGURE 1 - METHOD 3005A

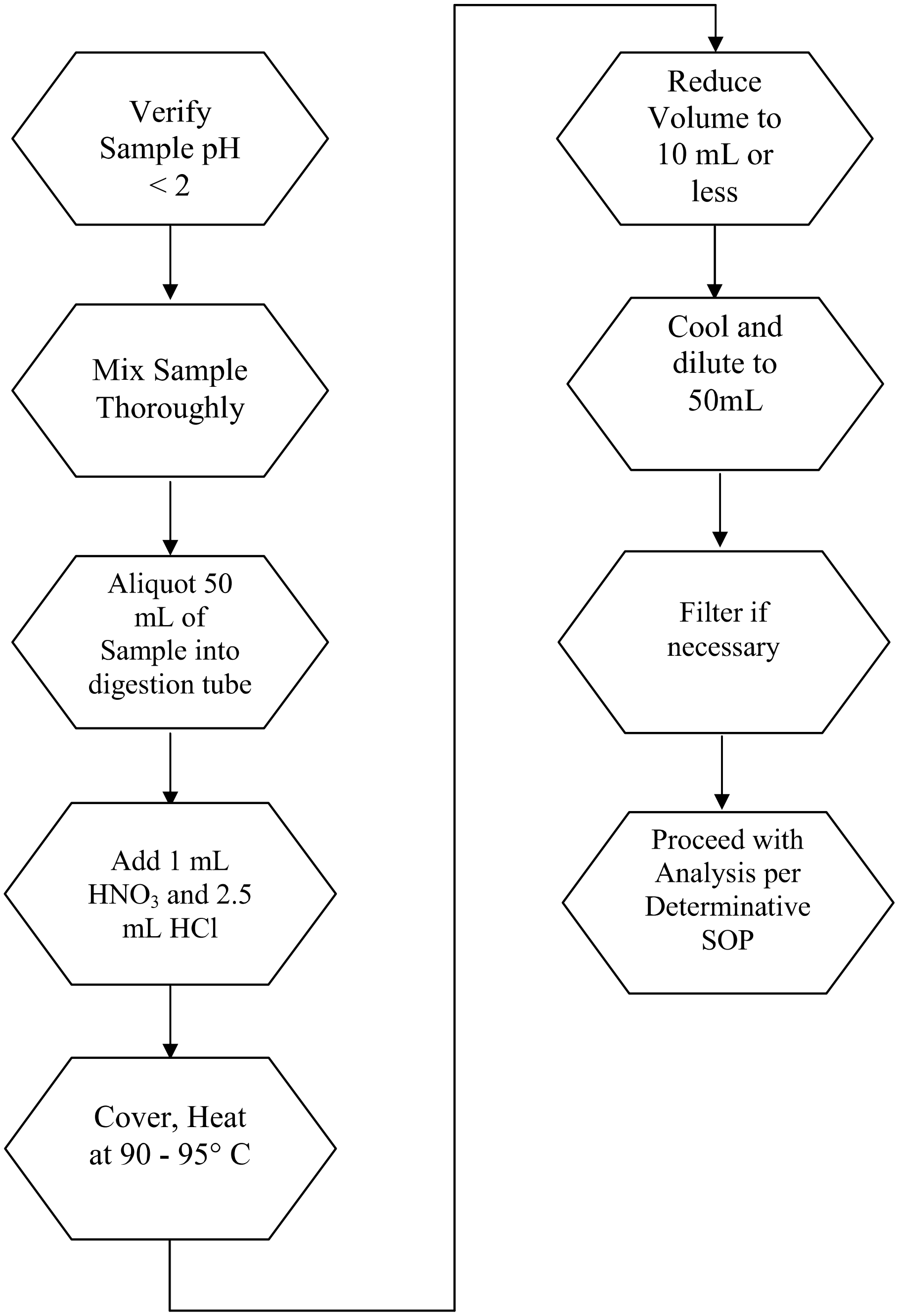


FIGURE 2 - METHOD 3010A

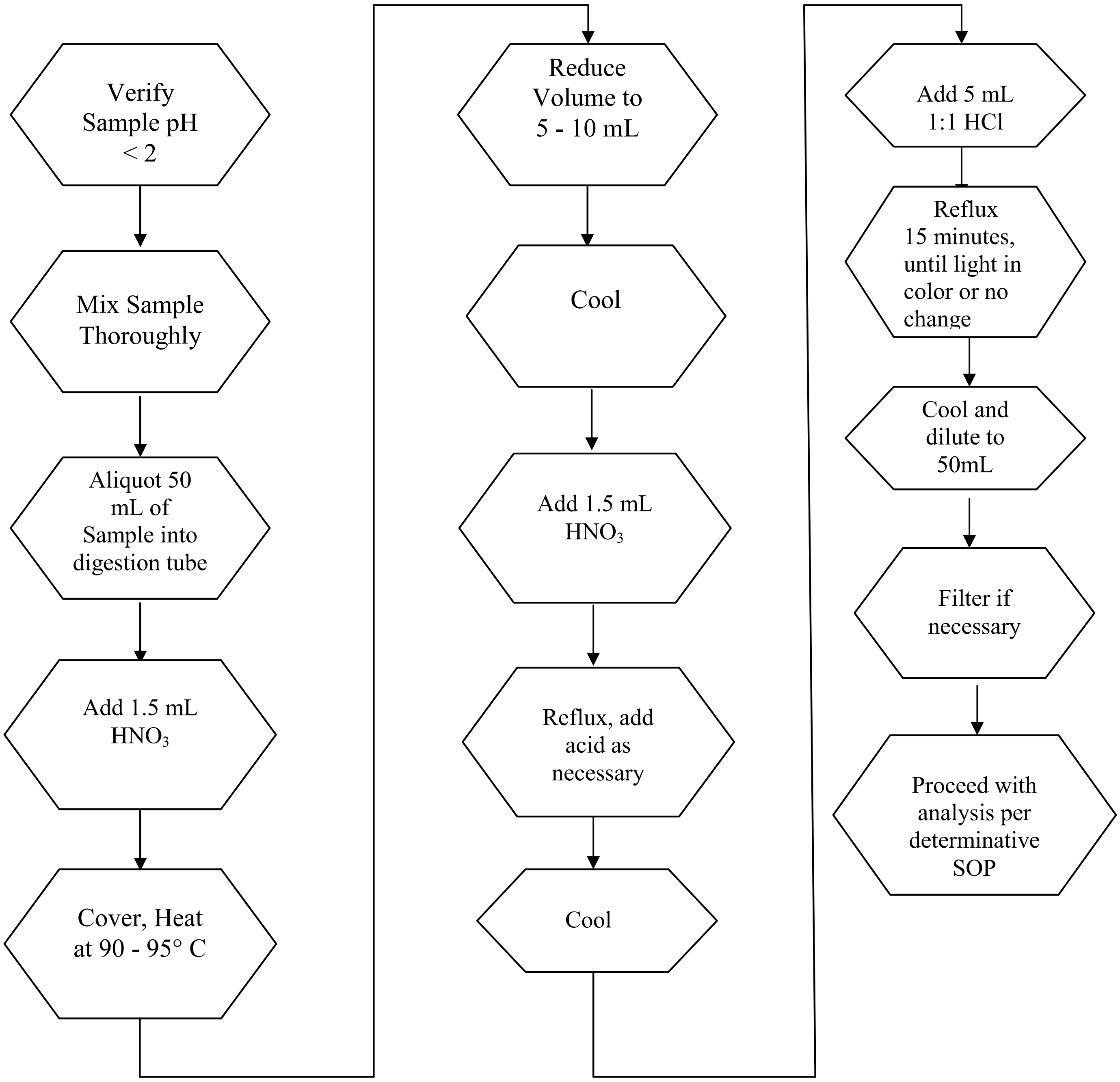


FIGURE 3 - METHOD 200.7

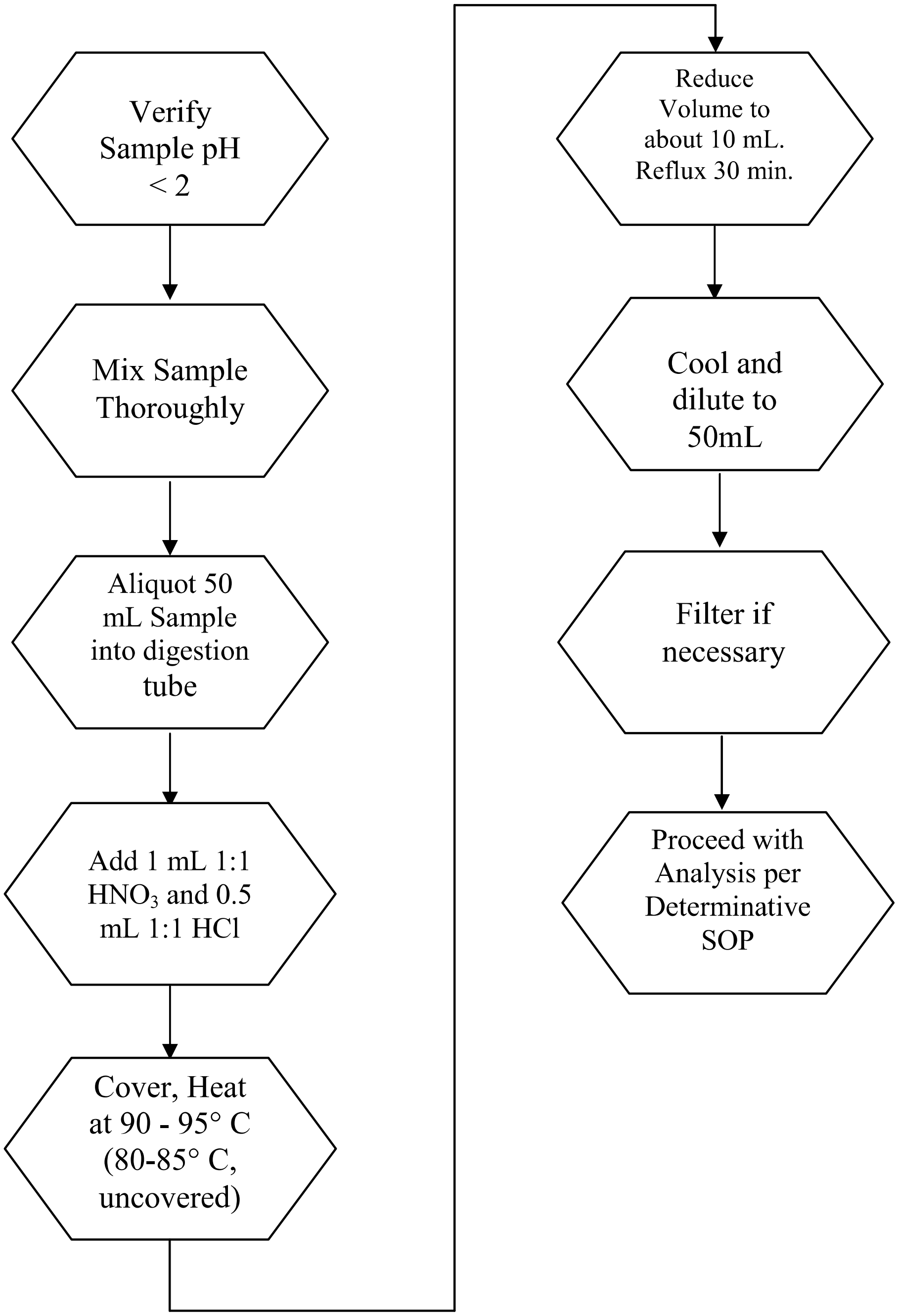


FIGURE 4 - METHOD 200.8

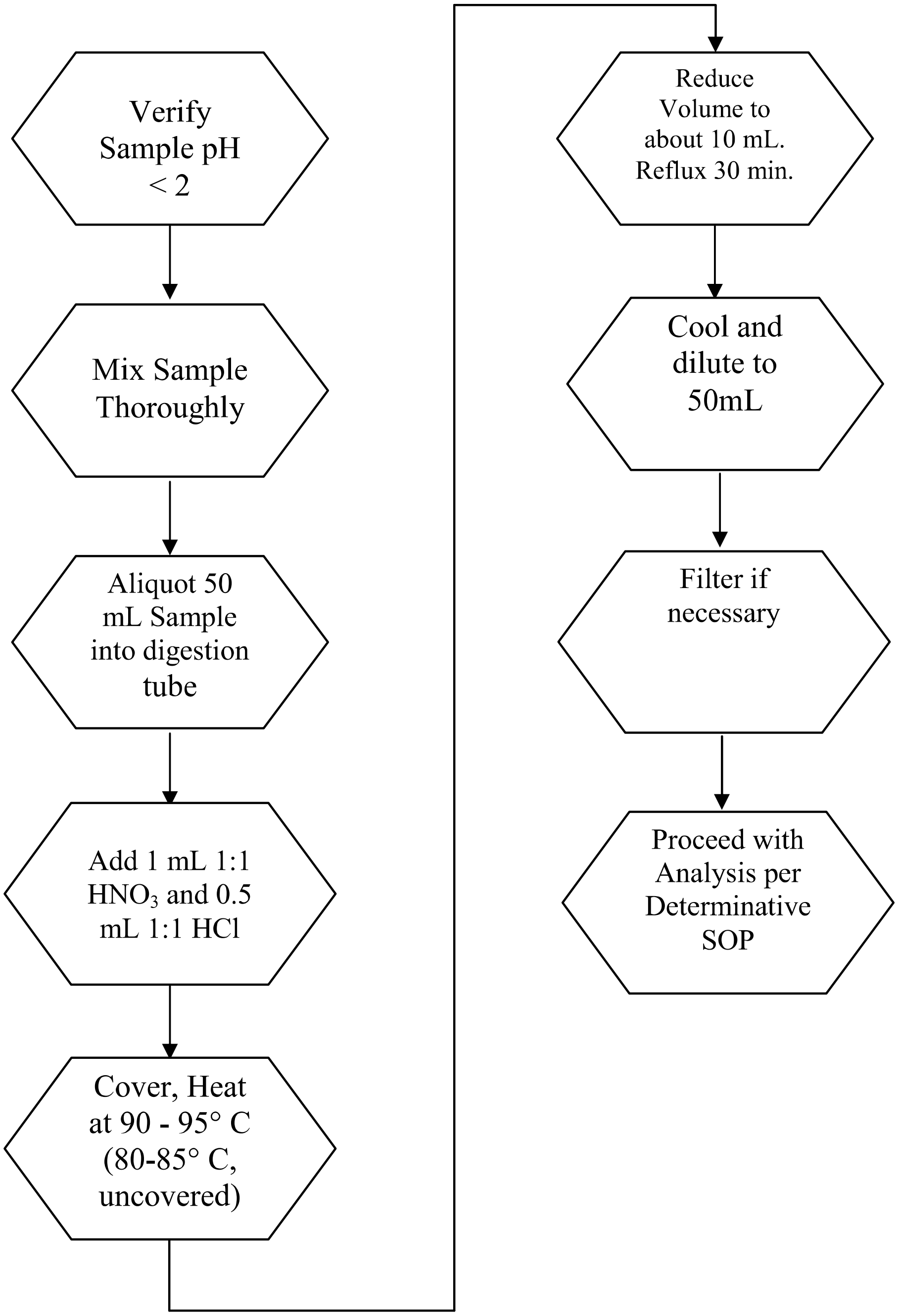
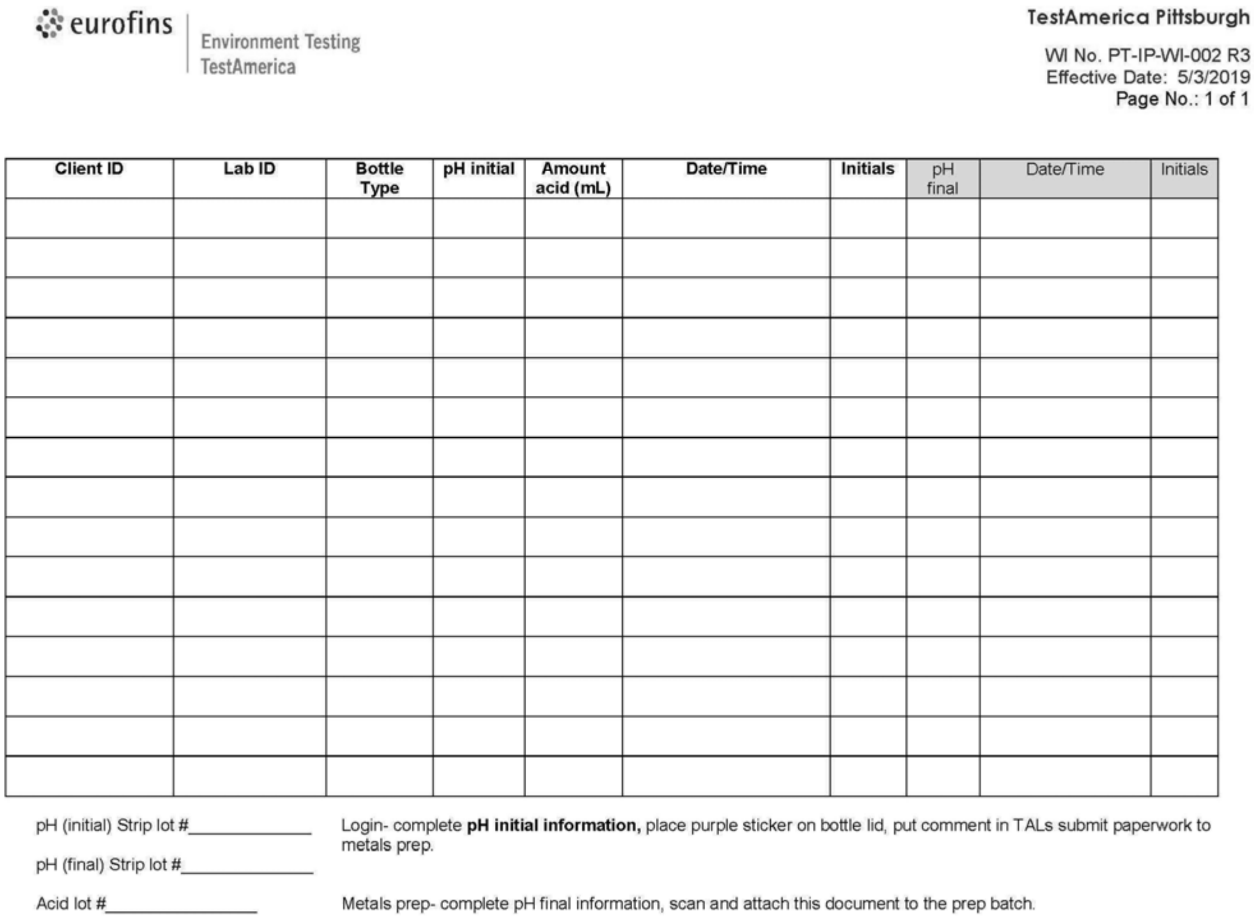


FIGURE 5 – EXAMPLE OF A LAB AQUEOUS PH PRESERVATION FORM



eurofins | Environment Testing
TestAmerica

TestAmerica Pittsburgh

WI No. PT-IP-WI-002 R3
Effective Date: 5/3/2019
Page No.: 1 of 1

Client ID	Lab ID	Bottle Type	pH initial	Amount acid (mL)	Date/Time	Initials	pH final	Date/Time	Initials

pH (initial) Strip lot #______________

pH (final) Strip lot #______________

Acid lot #________________

Login- complete **pH initial information,** place purple sticker on bottle lid, put comment in TALs submit paperwork to metals prep.

Metals prep- complete pH final information, scan and attach this document to the prep batch.

APPENDIX A

TABLES

TABLE I
Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 12.0 of the SOP are met.

TABLE II
Approved Preparation Method Analytes - NPDES

ELEMENT	Symbol	CAS Number	200.7
Aluminum	Al	7429-90-5	X
Antimony	Sb	7440-36-0	X
Arsenic	As	7440-38-2	X
Boron	B	7440-42-8	X
Barium	Ba	7440-39-3	X
Beryllium	Be	7440-41-7	X
Cadmium	Cd	7440-43-9	X
Calcium	Ca	7440-70-2	X
Chromium	Cr	7440-47-3	X
Cobalt	Co	7440-48-4	X
Copper	Cu	7440-50-8	X
Iron	Fe	7439-89-6	X
Lead	Pb	7439-92-1	X
Magnesium	Mg	7439-95-4	X
Manganese	Mn	7439-96-5	X
Molybdenum	Mo	7439-98-7	X
Nickel	Ni	7440-02-0	X
Potassium	K	7440-09-7	X
Selenium	Se	7782-49-2	X
Silicon	Si	7631-86-9	X
Silver	Ag	7440-22-4	X
Sodium	Na	7440-23-5	X
Thallium	Tl	7440-28-0	X
Vanadium	V	7440-62-2	X
Zinc	Zn	7440-66-6	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 12.0 of the SOP are met.

TABLE III
ICP/ICPMS Matrix Spike and Aqueous Laboratory Control Sample Levels

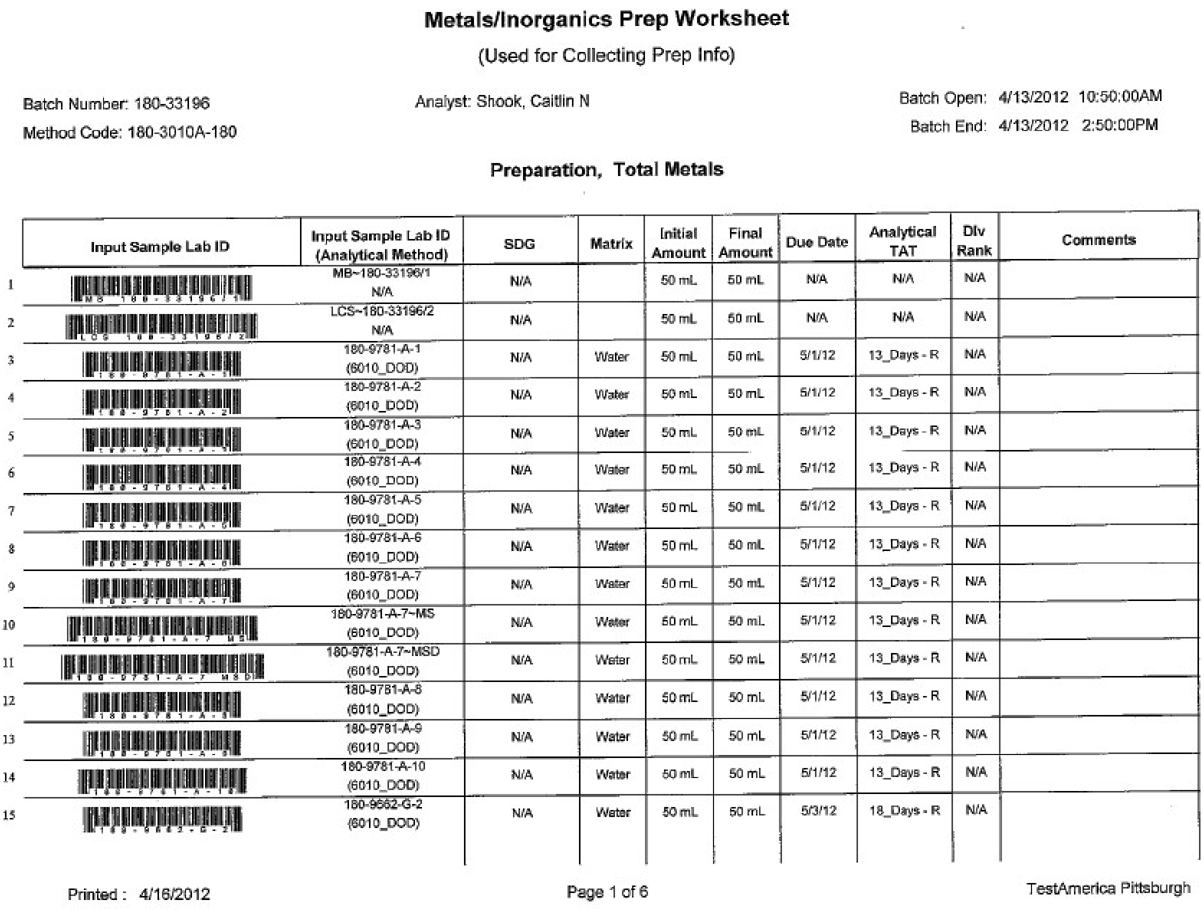
ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	1000	5000
Antimony	50	250
Arsenic	200	1000
Barium	200	1000
Beryllium	100	500
Cadmium	100	500
Calcium	5000	25000
Chromium	100	500
Cobalt	100	500
Copper	100	500
Iron	1000	5000
Lead	100	500
Lithium	100	500
Magnesium	5000	25000
Manganese	100	500
Molybdenum	100	500
Nickel	100	500
Phosphorous	1000	5000
Potassium	5000	25000
Selenium	200	1000
Silver	50	250
Sodium	5000	25000
Strontium	100	500
Thallium	200	1000
Vanadium	100	500
Zinc	50	250
Boron	250	1250
Silicon	200	1000
Tin	200	1000
Titanium	100	500
Thorium	0.05	50
Uranium	0.05	50

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.25 mL working spike (7.2) to 50 mL of sample.

TABLE IV
TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	2000	100000	20000
Cadmium	500	1000	500
Chromium	500	5000	2000
Lead	500	5000	5000
Selenium	500	1000	5000
Silver	500	5000	500

APPENDIX B – TALS METALS PREP EXAMPLE WORKSHEETS



Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 180-33196
Method Code: 180-3010A-180

Analyst: Shook, Caitlin N

Batch Open: 4/13/2012 10:50:00AM
Batch End: 4/13/2012 2:50:00PM

Preparation, Total Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments
1		MB~180-33196/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
2		LCS~180-33196/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
3		180-9781-A-1 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
4		180-9781-A-2 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
5		180-9781-A-3 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
6		180-9781-A-4 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
7		180-9781-A-5 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
8		180-9781-A-6 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
9		180-9781-A-7 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
10		180-9781-A-7~MS (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
11		180-9781-A-7~MSD (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
12		180-9781-A-8 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
13		180-9781-A-9 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
14		180-9781-A-10 (6010_DOD)	N/A	Water	50 mL	50 mL	5/1/12	13_Days - R	N/A	
15		180-9562-G-2 (6010_DOD)	N/A	Water	50 mL	50 mL	5/3/12	18_Days - R	N/A	

Printed : 4/16/2012

Page 1 of 6

TestAmerica Pittsburgh

APPENDIX C
CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

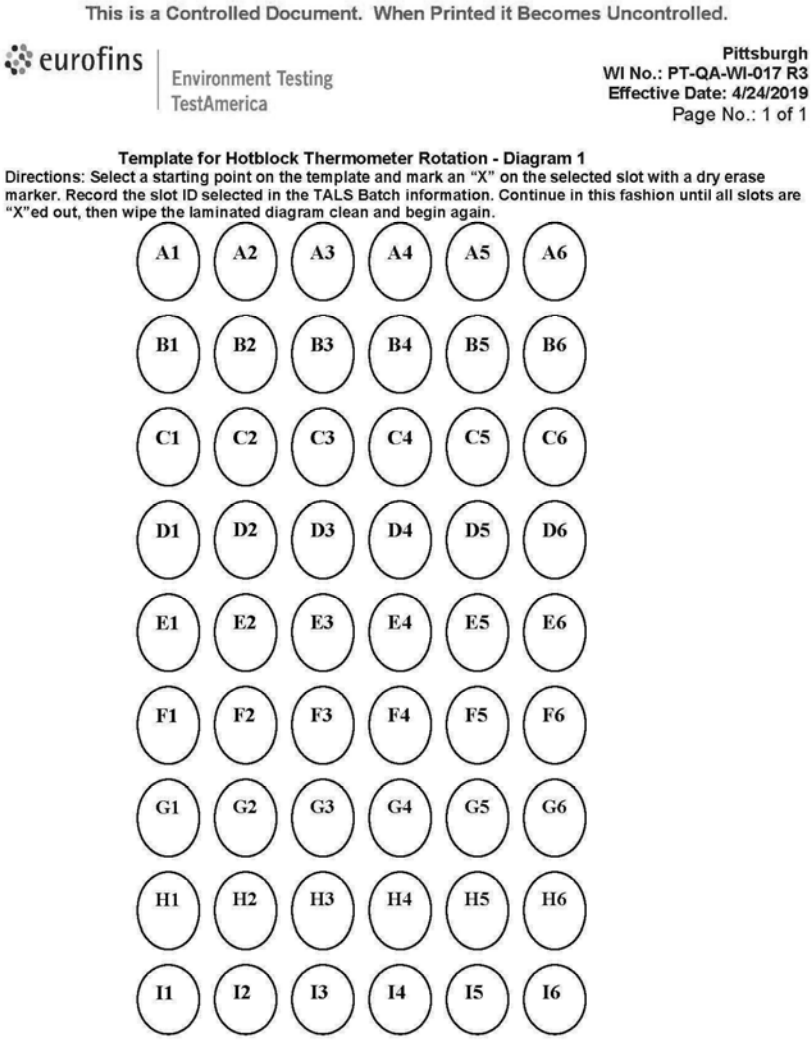
The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with nitric acid prior to routine cleaning.

APPENDIX D
Example of Template for Hotblock Thermometer Rotation



This is a Controlled Document. When Printed it Becomes Uncontrolled.

eurofins | Environment Testing
TestAmerica

Pittsburgh
WI No.: PT-QA-WI-017 R3
Effective Date: 4/24/2019
Page No.: 1 of 1

Template for Hotblock Thermometer Rotation - Diagram 1

Directions: Select a starting point on the template and mark an "X" on the selected slot with a dry erase marker. Record the slot ID selected in the TALS Batch information. Continue in this fashion until all slots are "X"ed out, then wipe the laminated diagram clean and begin again.

A1	A2	A3	A4	A5	A6
B1	B2	B3	B4	B5	B6
C1	C2	C3	C4	C5	C6
D1	D2	D3	D4	D5	D6
E1	E2	E3	E4	E5	E6
F1	F2	F3	F4	F5	F6
G1	G2	G3	G4	G5	G6
H1	H2	H3	H4	H5	H6
I1	I2	I3	I4	I5	I6

eurofins | Environment Testing
TestAmerica

Pittsburgh
SOP Change Form
PT-QA-WI-023 R2
Effective Date: 4/24/2019

SOP # with Revision: PT-MT-002, Rev. 17
SOP Title: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
Amendment Number: A1

Effective Date of Amendment:
Append this form to the front of the SOP copy.

CONTROLLED DISTRIBUTION
COPY # : **001**
ISSUED TO : **Pittsburgh SOP Directory**

The following SOP change is in effect as of the stated date. *This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record.*

1. Briefly explain the reason for the update:

   Revision of SOP Method Blank Acceptance Criteria and corrective actions

2. Category for SOP Change:

☐ Typographical Corrections (Non-Technical) – Retraining Not Required.
☐ Typographical Corrections (Technical- define below) – Retraining is required.
☒ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: [✓ No] / Yes: # pages attached = ___)

- Table in section 9.1, remove "Common lab contaminants may be accepted up to 5x the RL." from the Method Blank Acceptance Criteria.
- Change Section 9.2.1.2 to say "Results associated with MB's contaminated above the MDL will be reported with "B" qualifiers. Samples associated with MB's contaminated above the RL (or other project limit) must be reanalyzed along with the contaminated MB. If contamination in the MB is confirmed by the reanalysis, the MB and all associated samples must be redigested and reanalyzed.
  Remove bullets 1, 2, and 3 from this section and remove "none of the above situations applies, and" from the first sentence of the remaining bullet.

Does this change result in a Method Modification? Yes [✓ No]
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

7/20/2021
Approval Signature/Date: Technical

Amanda L Gulli 07/28/21
Approval Signature/Date: QA Manager or Designee

Title: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS)

Method(s): EPA 200.8, SW-846 6020, 6020A, 6020B

Approvals (Signature/Date):

	8/28/2020		8/31/2020
Roseann Ruyechan Inorganics Department Manager	Date	Steve Jackson Regional Safety Coordinator	Date
	8/26/2020		9/10/2020
Virginia Zusman Quality Assurance Manager	Date	Debbie Lowe Laboratory Director	Date

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. d/b/a Eurofins TestAmerica and its affiliates ("Eurofins TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Eurofins TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically provided. The user also agrees not to give access to this document to any third parties including but not limited to consultants, unless such third parties specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2020 TESTAMERICA LABORATORIES, INC. d/b/a EUROFINS TESTAMERICA ALL RIGHTS RESERVED.

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 6020, 6020A, 6020B and EPA Method 200.8.

1.2. This method is applicable to drinking, surface, and saline waters, soil, sediment, wipe, tissue and waste samples. (Use of the collision reaction cell technology is not allowed for drinking water analysis using method 200.8.)

1.3. The standard reporting limits and list of metals analyzed for by ICP-MS are listed in Table 1. Results below the standard reporting limit, but above the current method detection limit (MDL), may be reported and qualified as estimated.

1.4. Elements that may be determined using this procedure include: Ag, Al, Sb, As, Ba, Be, B, Ca, Cd, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, P, Se, Si, Sn, Sr, Th, Ti, Tl, U, V, Zn.

- Successful Ag analysis may require all solutions to be prepared as described, but with the addition of hydrochloric acid to 1% (v/v). This may degrade performance for As, Se and V.
- Collision Cell Technology (CCT) is used in analysis for Al, Sb, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Pb, Mg, Mn, Mo, Na, Ni, P, K, Sc, Se, Si, Sr, Tl, Th, Ti, U, V, and Zn.
    - CCT is not used when analyzing for B, Be, and Li.

1.5. Additional elements may be analyzed using this procedure, provide all appropriate QC measure are evaluated and found acceptable.

1.6. On occasion clients may require slight modifications to this SOP. Any modification to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM.

2. SUMMARY OF METHOD

2.1. Samples requiring preparation are processed as in PT-IP-002 (Acid Digestion of Soils by SW 846 Method 3050B), and PT-IP-003 (Acid Digestion of Aqueous Samples by SW 846 Methods 3005A, 3010A and EPA 200.7 and 200.8)

2.2. The sample solution is introduced into a pneumatic nebulizer via a peristaltic pump. The nebulizer generates a fine aerosol by bringing the solution into contact with a high velocity flow of argon gas at its tip. The nebulized sample is sorted by droplet size in the spray chamber. Large droplets are rejected, while smaller particles are transported with the gas stream into the plasma.

2.3. The argon plasma operates with a continuously applied radio frequency (RF) field to give a high-energy discharge consisting of argon atoms, ions and electrons. The hottest part of the plasma can attain 6000-8000 K. In the plasma, aerosol droplets undergo evaporation, atomization and ionization. Ions are sampled through an aperture in a metal cone (sampler) at atmospheric pressure, into the expansion region at about 2 mbar and subsequently through an aperture in a second metal cone (skimmer) into the intermediate chamber.

2.4. An electrostatic ion lens system focuses the ion beam through a differential aperture into the analyser chamber, at about 10-7 mbar. The ions are filtered by mass-to-charge ratio in microsecond timescales by the quadrupole. The selected mass is detected by a discrete dynode electron multiplier. The multiplier has two simultaneous modes of operation: pulse count and analogue. The combination of these two modes allows seamless detection spanning 8 - 9 orders of magnitude. A detector "cross-calibration" is required for the analogue counts to be converted to equivalent pulse counts. The output from the detector is proportional to the concentration of the element in the aspirated solution, hence the concentration of unknown samples may be calculated when the instrument response is calibrated with standards of known concentration.

2.5. The linear range may vary from instrument to instrument and is dependent upon the sensitivity determined by the optimization parameters. This is determined by the individual laboratory and instrument.

2.6. Calibration standard concentrations are listed in Table 2 at the end of this document.

3. DEFINITIONS

3.1. TALS – Eurofins TestAmerica Laboratory Information Management System

3.2. NCM – Non-Conformance Memo – a system within TALS for the lab to communicate when there is an anomaly seen with samples or a batch or QC failure.

3.3. Please refer to the glossary in the Laboratory Quality Assurance Manual (PT–QA-M-001) for definitions of general terms.

4. INTERFERENCES

4.1. Isobaric interferences. Elemental isobaric interferences occur when different elements have isotopes at the same nominal mass, e.g. ^{114}Cd and ^{114}Sn. Problematic elemental isobaric interferences for these methods are listed in Table 10. The correction factors given in Table C are based on theoretical isotopic abundance ratios and may require adjustment.

Table C - Isobaric Interferences and Correction Equations

m/z	Element	Interferent	Correction
58	Ni	Fe	58Ni=58M-0.0040*56Fe
64	Zn	Ni	64Zn=64M-0.0440*60Ni
78	Se	Gd	78Se=78Se-(0.06*154Gd)
82	Se	Kr	82Se=82M-1.0010*83Kr
114	Cd	Sn	114Cd=114M-0.0270*118Sn
115	In	Sn	115In=115M-0.0140*118Sn
123	Sb	Te	123Sb=123M-0.1240*125Te
138	Ba	Ce	138Ba=138M-0.0030*140Ce

4.2. Abundance Sensitivity - Abundance sensitivity is the ability of the quadrupole to separate a low intensity peak from an adjacent high intensity peak. An example of the requirement of this is the detection of low concentrations of manganese (m/z 55) in the presence of high concentrations of iron (m/z 56). Quadrupole resolution and bias can be adjusted during set-up to resolve these signals.

4.3. Isobaric Polyatomic Ion Interferences - Polyatomic ions are produced by chemical reaction in the plasma and the interface region. If these polyatomic ions have the same nominal mass to charge (m/z) ratio as an element a polyatomic interference is observed. Some of the correction factors given in Table D are based on theoretical isotopic abundance ratios and may require adjustment. Other factors were derived empirically. The stability of the empirical factors was determined by the manufacturer. The factors require little or no adjustment and can be transferred between similarly configured instruments. These interferences can also be eliminated by use of Collision Cell Technology (CCT). This technology introduces a 93% Helium and 7% Hydrogen gas into the instrument before the sample reaches the quadruple. This gaseous mixture collides with the polyatomic formed bonds to break them. For example, the gaseous mixture will break the ArCl (Mass 75) bond leaving Ar^{40} and Cl^{35} as separate masses, therefore the only element detected at Mass 75 is arsenic.

Table D - Isobaric Polyatomic Interferences and Correction Equations

m/z	Element	Interferent	Correction
51	V	ClO	51V = 51M-3.0460*53ClO 53ClO = M53-0.114*52Cr
52	Cr	ArC, ClOH	52Cr = 52M-0.0050*13C
56	Fe	CaO	56Fe = 56M-0.1500*43Ca
56	Co	CaO, CaOH	59Co = 59M-0.0046*43Ca
60	Ni	CaO	60Ni = 60M-0.0020*43Ca
75	As	ArCl	75As = 75M-3.000*77ArCl 77ArCl = 77M-0.8000*82Se 82Se = 82M-1.0010*83Kr
111	Cd	MoO	111Cd = 111M-0.9820*108MoO 108MoO = 108M-0.712*106Cd

4.4. Physical Interferences - Physical interferences include transport effects, ionization effects and deposition effects in the sample introduction system, plasma and interface, which result in signal suppression and signal drift. Transport effects arise from variations in solution properties, e.g. viscosity or surface tension, which affect nebulization efficiency and aerosol droplet size. The concentration of dissolved matter will affect the ionization efficiency of the elements in the plasma and will cause a mass-dependant suppression effect and contribute to space-charge effects. Dissolved matter may also condense on the cones, altering the ion beam profile. This normally manifests itself as a time-dependant downward signal drift. To reduce the severity of these effects it is advised that the total dissolved solids concentration of solutions aspirated should be limited to <0.05%. Samples known to contain higher dissolved solids concentrations should be diluted. Signal suppression and drift can be corrected, to a degree, with the use of internal standardization techniques. Since these effects can be mass-dependant and may be related to the ionization potential of the element, a multiple-element internal standard approach should be used.

4.5. Memory Effects - Memory effects occur when the signal for an element from a sample contributes to the signal of a subsequent sample. This effect can be severe for certain elements due to their physico-chemical properties, e.g. mercury. This effect is minimised by aspirating a wash solution between samples. A monitored wash can be used in order to ensure that element signals recover to the background level.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-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.

5.2. Specific Safety Concerns or Requirements

5.2.1. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3. 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 SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

Table E – Known Hazardous Materials

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. The waste pumped from the spray chamber is corrosive and must be handled with care, especially if large volume containers are used, as these may be heavy and awkward to carry. Empty the waste vessel daily to reduce the quantity that must be disposed each time and to keep weight to a minimum. Protective clothing, including hand and eye protection must be worn when handling this waste.

5.6. The wash solution is corrosive and must be handled with care. This solution must be prepared and stored in a vessel made of a robust acid-resistant material with a tight fitting lid that it is resistant to breakage if dropped. Large volumes of this solution will be heavy and may be awkward to carry. Ensure adequate provision for transporting the vessel, i.e. suitable handles on the vessel, minimum distance between the preparation area and the instrument. Use a cart to transport the vessel where necessary or ask for assistance in carrying.

5.7. Many of the concentrated metal standard solutions are toxic and must be handled with care. Skin and eye protection should be worn when handling and inhalation of vapours must be prevented.

5.8. Fumes generated by the plasma can be hazardous and must be removed from the laboratory with an extraction system designed to continuously remove the fumes generated by the instrument. If the extraction system is faulty do not attempt to use the instrument. The extraction system should be inspected on a regular basis.

5.9. The plasma is a source of radio frequency (RF) radiation and intense, ultra-violet radiation that can damage the eyes. This radiation is normally contained by the system, but operators must be aware of the dangers. The instrument must be properly maintained by qualified service personnel. Never attempt to defeat hardware interlocks – they are there for your safety.

5.10. Should the plasma need to be extinguished in an emergency, open the torch box door. This will immediately cut-off the power to the plasma RF generator, extinguishing the plasma. After extinguishing the plasma, the torch, torch box, cones and cone housing may remain very hot for some time. Operators must be aware of this fact and allow cooling time prior to handling these components.

5.11. There are high voltage components inside the instrument. Routine maintenance does not require access to any of the electronic components. If an electronic fault is suspected, a qualified service engineer must be called. Do not attempt to tamper with electronic components yourself.

5.12. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.13. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.14. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EHSC.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Instrumentation

6.1.1. Agilent 7800 ICP-MS

- SPS 4 autosampler with ISIS 3
- ICP-MS MASS HUNTER Software, version 4.6 C.0.106

6.1.2. Agilent 7900 ICP-MS

- SPS 4 Autosampler with ISIS 3
- ICP-MS MASS HUNTER Software, version 4.2 C.0.102 or 4.5 C.0.105

6.2. Supplies

6.2.1. Ultrapure water system capable of delivering de-ionized, polished water of at least 18 MΩ/cm

6.2.2. Instrument specific pump tubing or required diameters

6.2.3. A range of adjustable pipettes, such as Rainin pipettes. Adjustable pipettes with a capacity of 0.1 mL, 1 mL, and 10 mL are recommended. These must be calibrated regularly to ensure accurate volumes are delivered.

6.2.4. Volumetric flasks, Class A, various volumes

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard. Quantities different than what is specified can be made provided that the ratios remain the same.

7.1. Laboratory Water - All laboratory water used in these procedures must be of very high quality, purified with a reverse osmosis system and polished with an ion exchange system to give a final product of resistivity >18 MΩ/cm.

7.2. Hydrochloric Acid (Conc.) - Hydrochloric acid must be at least J.T. Baker “Instra Analyzed”(9530-33), EMD “Omnitrace” (HX0607), or Fisher “Optima” - grade or equivalent.

7.3. Nitric Acid (Conc.) - Nitric acid must be at least J.T. Baker “Instra Analyzed” (9598-34), or Macron “AR Select” (6623-46)

7.3.1. 2 % (m/v) Nitric Acid - This reagent is used for the calibration blank, ICB, CCB, sample dilution and solution preparation. Add 5 mL of Conc of HNO3 to laboratory water in a 250 mL volumetric flask and dilute to volume. Prepare solution fresh daily.

NOTE: Always add acid to water.

7.4. Composition of standards is given in Tables 2 through 9. All standards are purchased prepared and certified at the concentrations listed. The ICV standard is purchased from a different source than the standard used for initial calibration. The storage conditions and expiration dates provided by the manufacturer must be followed.

7.5. Standard and reagent use and preparation must be documented in the TALS AD batch and reagent module.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Water	250 mL Plastic bottle	100 mL	pH <2 preserved with HNO_3	180 days from collection	40 CFR Part 136
Soil/Wipes	4 oz. clear glass jar	2 grams	Cool to >0 but ≤ 6℃	180 days from collection	40 CFR Part 136
Tissues	4 oz. clear glass jar	2 grams	Frozen until ready to prepare	180 days from collection	40 CFR Part 136
Sediments	4 oz. clear glass jar	100 grams	Cool to >0 but ≤ 6℃	180 days from collection	40 CFR Part 136

8.1. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure. Method 200.8 has its own digestion specifications that are followed by the laboratory. Method 3005A is used for total recoverable metals, total metals and dissolved metals and method 3010A (3005A for samples from WV requiring Ag) is used for TCLP metals by 6020 and 6020A. These are covered in the SOP PT-IP-003. Upon consultation with the client dissolved samples can forego digestion, to help prevent contamination when very low detection limits are required.

8.2. Soil, wipe, tissue and waste samples should be digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure. The SOP for 3050B is PT-IP-002.

8.3. Dissolved metals samples that are filtered directly into a nitric acid preserved container may be digested immediately following the filtration. Samples cannot be filtered for dissolved metals after preservation.

9. QUALITY CONTROL

9.1. Summary of Quality Control Requirements

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Blank plus Single-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
Linear Range Standard - High Level Verification (Method 6020B only)	Daily after Initial calibration	±10% of the true value	Terminate analysis; Correct the problem; Recalibrate.
ICV	Beginning of every analytical run after ICAL	90 - 110 % Recovery	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
RLV/CRI/LLICV/LLCCV (ICVL or CCVL in TALS)	Beginning of every analytical run, immediately following the ICB. Method 6020A also requires analysis at the end of the analytical sequence.	50–150% Recovery for 200.8 & 6020 70–130% Recovery for 6020A 80 – 120% recovery for 6020B	Terminate analysis; Correct the problem; Recalibrate
CCV	Every 10 injections and at the end of the run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB
ICSA	Beginning of every run	For RL <10, results ±3x RL, For RL >10, results ± RL, or result ±20% of True Value [1]	Terminate analysis; Correct the problem; Recalibrate.
ICSAB (SIC for Method 6020B)	Immediately following each ICSA.	80 - 120% Recovery.	Terminate analysis; Correct the problem; Recalibrate.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to ± RL. (Common lab contaminants may be accepted up to 5x the RL.)	Re-digest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Method 200.8: 85 - 115% Recovery 6020/6020A/6020B: 80-120% Recovery	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation	Method 200.8: 70 – 130%	In the absence of client specific

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
	batch of up to 20 samples (6020/6020A/6020B) or one per every 10 or fewer samples (200.8).	Recovery 6020/6020A/6020B: 75-125% Recovery	requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 10.16.
Matrix Spike Duplicate	See Matrix Spike	Recovery limits same as MS RPD ≤ 20% for all methods	See Corrective Action for Matrix Spike.
Post-Digestion Spike	Only performed when specific elements failed original matrix spike limits and only if spike concentration added was > the concentration determined in unspiked sample.	75-125% Recovery (6020/6020B) 80-120% Recovery (6020A)	Report and qualify parent sample results as estimated.
Dilution Test	One per prep batch	For samples ≥ 50x MDL, dilutions must agree within 10% (200.8/6020/6020A). For samples ≥ 50x MDL, dilutions must agree within 20% (6020B).	Narrate the possibility of physical or chemical interference per client request.
Internal Standard	In all samples and QC	(compared to intensities in the calibration blank) 60-125% (200.8) 30-120% (6020) Samples 30-150%; QC 70-150% (6020A)[2] 30-150% (6020B)[2]	Dilute sample and reanalyze

[1] For batches containing samples from Wisconsin, the ICSA must be within ±RL for all non-spiked elements

[2] Note: All samples from South Carolina must adhere to Internal Standard Response limits of 70-130% for samples and QC for methods 6020A and 6020B.

9.2. Batch QC

9.2.1. Method Blank (MB)

The method blank consists of reagent water, or Teflon chips for non-aqueous matrix batches, containing all reagents specific to the method that is carried through the entire preparation and analysis procedure with the samples. 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 element concentrations or false positive data.

9.2.1.1 MB must not contain any target elements at or above the Reporting Limit (RL), or more negative than RL. Certain programs may require a more stringent evaluation of the method blank, for instance, that the blank not contain any elements of interest at a concentration greater than ½ the reporting limit or greater than the MDL. Check projects for specific criteria.

9.2.1.2 Results associated with MB's contaminated above the MDL will be reported with B flags in TALS. Samples associated with MBs contaminated above the RL (or other limit per project specifications) must be reanalyzed along with the failing MB for the affected elements. Reanalysis is not required if one of the following situations exists.

- If the element is a common laboratory contaminant (copper, iron, zinc), the data may be reported with qualifiers if the concentration of the element in the MB is less than 5 times the RL. The "Method Blank – Report, Common Lab Contaminant <5x RL" NCM must be used when reporting such data as well as the qualifier flag.
- If the MB is contaminated above the RL, and associated sample concentrations are >10 times the MB concentration, results may be reported using the "Method Blank – Report, 10X" NCM.
- If the contaminating element is not detected above the RL in samples associated with an unacceptable MB, results for those samples may be reported using the "Method Blank – Report, ND" NCM.
- If none of the above situations applies, and reanalysis is not possible due to sample volume or other problem, the project manager must be notified and the client contacted with the details. If results are to be reported, they must be properly qualified and the nonconformance described in the case narrative.

If contamination in the MB persists in the reanalysis then the entire batch will be redigested and reanalyzed.

9.2.2. Laboratory Control Sample (LCS)

The LCS consists of reagent water, or Teflon chips for non-aqueous matrix batches, spiked with the elements listed in Appendix 6, unless otherwise requested by the client. The LCS is carried through the entire preparation and analysis procedure with the samples, and is used to monitor for bias and method performance with each batch. Results must be within 85-115% of the true value for method 200.8, or 80-120% for methods 6020/6020A/6020B.

9.2.2.1. If any element in the LCS is outside the control limits, corrective action must occur:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and all associated samples, and if still outside of control limits,

- Re-digest and reanalyze all samples in the QC batch.

9.2.2.2. Re-digestion/reanalysis is not required if recovery of the LCS is above the control limits, up to 140%, and the affected element is not detected above the RL in the associated sample. Results may be reported using the LCS/LCSD - %R, High" NCM.

9.2.2.3. If the above criterion is not met, and reanalysis is not possible, the project manager must be notified and the client contacted with the details. If results are to be reported, they must be properly qualified and the nonconformance described in the case narrative.

9.2.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The MS and MSD are spiked into separate portions of a client sample prior to any digestion steps, and are carried through all the preparation and analytical steps along with the samples. Samples for MS/MSD are chosen at random from all client samples in the batch, unless a client has specified a sample to be used. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.2.3.1 Some programs require a Matrix Duplicate (MD) and Matrix Spike (MS) in lieu of an MS/MSD. When a MD/MS is performed the MS is evaluated for accuracy (% recovery) and the MD is evaluated for precision (RPD). Percent recovery must be within 70-130% for method 200.8 and 75-125% for methods 6020/6020A/6020B. The %RPD must be $\leq$20% for all methods.

9.2.3.2 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 element in the Laboratory Control Sample (LCS). If the recovery of the element in the LCS is within limits, then the laboratory operation is in control and results may be reported with the parent sample and MS/MSD (or MD) qualified and the nonconformance described in the case narrative.

- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the process is out of control and corrective action must be taken. Corrective action will include re-digestion and reanalysis of the batch.
- If the concentration of the element in the parent sample is greater than 4 times the amount of spike added, then routine control limits may not apply. Recoveries are still evaluated, however reanalysis is not required. Results are reported with qualifiers and the condition described in the case narrative.

9.2.3.3 If insufficient volume of samples is received to perform an MS/MSD analysis with a batch, an LCS/LCSD may be performed, and the lack of volume documented by attaching an "MS/MSD/DUP – Insufficient Volume" NCM to the batch in TALS.

9.2.4. Post-Digestion Spike Samples (PDS)

For methods 6020 and 6020B, a post digestion spike will be run on a sample if the MS/MSD for recovery falls outside of 75-125%. For method 6020A, recovery must be between 80-120%. A PDS is not required for method 200.8.

9.2.4.1 A post digestion spike is a spike added to the already digested portion of the parent sample. (The spike solutions from the digestions section are used for spiking. See SOP PT-IP-002 or PT-IP-003 for details of these solutions.)

9.2.4.2 If the PDS also fails, then matrix effects are confirmed. Results will be reported using the “Post Digestion Spike - %R” NCM.

9.2.5. Serial Dilution Samples (SER)

Some regulatory programs require a dilution test to be performed for each matrix within an analytical batch determination. If the element concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution – or 50x before dilution), an analysis of a 1:5 dilution should be performed. Only elements for which the original sample result is $\geq$50x the MDL are evaluated.

9.2.5.1 The results of the dilution (corrected for the dilution factor) must agree with the original sample results within 10% (20% limit for method 6020B).

9.2.5.2 If the %RPD is above the control limit, a matrix interference may be indicated. Report results along with the “Serial Dilution - %D” NCM.

9.3. Instrument QC

9.3.1. Mass Spectrometer Tuning Check must be performed daily prior to calibration.

- Peak resolution must be < 0.90 amu at 10% peak height for methods 200.8 and 6020/6020A/6020B.
- Mass calibration must be within $\pm$ 0.1 amu from the actual value for the 6 tuning elements (Be, Ce, Co, In, Mg and Pb).
- For method 200.8, peak width must be approximately 0.75 amu at 5% peak height.
- See section 10.2.2 for additional details.

9.3.2. Initial Calibration is performed daily using a single calibration standard and a blank for each element. Refer to Table 2 for standard concentrations.

9.3.3. Linear Range Verification (LR) - The linear range is determined semi-annually (2x/year) for each element on the standard list. For method 6020B, the upper linear range must be verified daily with a standard run at the highest reportable concentration. Recovery of the daily standard must be within 90-110% of the true value. See Section 12 for details of the linear range verification.

9.3.4. The internal standard (IS) intensities in samples must be within 60 to 125% of the IS intensities for the Calibration Blank for method 200.8, 30% to 120% for method 6020, and 30 to 150% for method 6020B. For method 6020A, Internal Standard response must be 70 to 150% for QC (ICV/CCV, ICB/CCB, MB, LCS) and 30 to 150% for samples. (Note: For samples from South Carolina, Internal Standard Responses must be within limits of 70-130% for samples and QC for methods 6020A and 6020B.) If this criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. IS failures must be documented using the "ISTD – Matrix, DL Required" NCM.

- For method 6020 the internal standard intensity in the ICV, ICB, CCV and CCB should be within 20% of the IS intensity in the calibration blank of the initial calibration. If not, the analyst should check for any instrument anomalies and continue if none are noted.
- For methods 200.8, 6020A, and 6020B the IS acceptance range for calibration verification does not vary from the limits noted above.

9.3.5. Interference Check Solutions - ICSA and ICSAB (SIC for method 6020B) - The "true" value for non-spiked elements will be taken as zero, unless otherwise indicated in the solution manufacturer's literature. The software automatically checks for compliance with the limits below.

9.3.5.1. Results for non-spike elements with RL <10 ug/L must be within ± 3 time the RL of 0. For non-spiked elements with RL ≥ 10 ug/L, results must be within ± the RL of 0. Spiked elements must be recovered within 80-120% of the certified value.

NOTE: For batches containing samples from Wisconsin, the ICSA must be within ± RL of 0 for all non-spiked elements.

9.3.5.2. If a result falls outside this range, the analysis must be terminated and the samples associated must be reanalyzed.

9.3.5.3. See Tables 8 and 9 for current certified values for these solutions.

9.3.6. Initial Calibration Verification (ICV) - Calibration accuracy is verified by analyzing a second source standard (ICV). Recoveries for the ICV must be within 90-110% of the true value. If ICV recovery is outside control limits, determine the source of the failures and repeat the Initial Calibration and ICV analysis.

9.3.7. Initial Calibration Blank (ICB) - An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. Certain programs, may require a more stringent evaluation of ICB, for instance, that the blank not contain any elements of interest at a concentration greater than ½ the reporting limit or greater than the MDL.

9.3.8. CRQL Check Standard (CRI/LLICV/LLCCV) - The CRQL/CRI/LLICV/LLCCV check is an undigested standard prepared at the RL and analysed daily for all methods. The standard must be analyzed at the beginning

(200.8/6020/6020A/6020B) and end (6020A only) of the analytical sequence. This standard is labelled as an ICVL or CCVL in TALS.

9.3.8.1. Recovery must be within 50-150% for methods 200.8 and 6020, and within 70-130% for method 6020A, and 80-120% for method 6020B.

- If any element is outside the range indicated, the standard may be re-run once. If the results fall within the required values upon re-run, no further corrective action need be taken. If still outside the acceptable range, the analysis shall be terminated, the problem corrected and the affected samples reanalyzed.

- For 6020A, if the closing CRI recovers high and outside of criteria, the sample results may be reported if sample concentrations are ≥10X the CRI concentration or < the RL.

9.3.9. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard (CCV) and blank (CCB).

9.3.9.1 CCV recovery must be within 90-110% of the true value. If outside this range, the analysis must be terminated, the problem corrected and the samples since the last valid CCV must be re-analyzed.

- If an element recovers >110% in the CCV, up to 120%, and the element is not detected above the RL in any bracketed sample, sample results may be reported with qualifier and using the “CCV - %D, High, Sample ND” NCM.

9.3.9.2 The CCB result must fall within ± RL from zero, unless all bracketed samples have concentrations ≥10X the CCB concentration or concentrations < the RL.

- Certain programs, may require a more stringent evaluation of the CCB, for instance, that the blank not contain any elements of interest at a concentration greater than ½ the reporting limit. The analyst should refer to the project notes provided by the PM to identify when this is an issue and if so what the corrective actions to take for exceedances.

9.3.9.3 Sample results may only be reported when bracketed by valid CCV/CCB pairs, unless one of the situations described above exists. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

10. PROCEDURE

10.1. Sample Preparation

10.1.1. Refer to SOPs PT-IP-002 (Acid Digestion of Soils by SW 846 Method 3050B) and PT-IP-003 (Acid Digestion of Aqueous Samples by SW 846 Methods 3005A, 3010A and EPA 200.7 and 200.8) for sample digestion procedures.

10.2. Calibration

10.2.1. Instrument start-up

Follow the instrument start-up procedure outlined in the Agilent ICP-MS 7800/7900 Operator’s Manual.

10.2.2. Instrument Tuning

10.2.2.1. Aspirate a 20 ppb tuning solution containing all of the tuning elements. The 6020/6020A tuning elements are Li, Co, In, and Tl. The instrument manufacturer monitors Mg, Ce, Be & Pb for instrument performance.

10.2.2.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.2.2.3. Resolution must be < 0.90 amu at 10% peak height for the 6 tuning elements (Be, Ce, Co, In, Mg, & Pb) for all methods, and for method 200.8, peak width must be approximately 0.75 amu at 5% peak height.

10.2.2.4. Mass calibration must be within ± 0.1 amu from the actual value for the 6 tuning elements (Be, Ce, Co, In, Mg, & Pb) or the mass calibration must be adjusted.

10.2.2.5. A “daily” performance check must be performed. This uses the same tuning solution as above with 5 replicates. The 6 tuning elements must have RSDs below 5%. The oxides ratio must be below 5%. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.2.2.6. Recommended analytical isotopes and additional masses that maybe monitored are given in Table 11. Recommended isotopes and additional masses that may be monitored are given in Table 12.

10.2.3. Initial Calibration

10.2.3.1. Calibration consists of a blank and the following calibration standards (STD1, STD 2X, and STD 3X see Table 2 for concentrations) in accordance with the manufacturer’s procedure. Use the average of three integrations for both calibration and sample analyses.

10.2.3.2. Following the STD, STD2X & STD3X, a Linear Range Verification (6020B only) and second source ICV and ICB are analysed.

10.2.3.3. For 6020/6020A/6020B, following the ICV/ICB pair, the CRI/RLV is run before the ICSA is analyzed. The CRI/RLV is analyzed again at the end of the sequence for 6020A.

10.2.3.4. For 6020, 6020A, and 6020B following the ICSA, analyze the ICSAB. The ICSAB must be within ± 20% of the true value. For method 200.8 ICSA and ICSAB is also analyzed although not required by the method.

10.2.3.5. Internal standards are added to all standards and samples by the instrument automatically during analysis.

10.2.4. Continuing Calibration

10.2.4.1. Following every 10 injections (including lab QC), analyze a CCV/CCB pair. These must be within ± 10% of the true value for analysis to continue. For methods 6020/6020A/6020B, a CCV/CCB pair should also be analyzed immediately after the ICSAB.

10.2.4.2. All samples must be bracketed by an acceptable CCV/CCB pair. Where a CCV/CCB fails the samples preceding it back to the last acceptable CCV/CCB must be reanalyzed.

10.2.5. Instrument Set-up

10.2.5.1. The instrument is configured a glass concentric nebulizer, glass impact bead spray chamber and a one-piece torch with 1.5mm ID injector tube. A Peltier spray chamber cooling unit is optional. Ensure that the interface cones and peristaltic pump tubing are fitted as required by the manufacturer.

10.2.5.2. Perform the daily maintenance as outlined in the instrument manual.

10.2.5.3. Switch the instrument into the operational state. During the automated ignition sequence, the following processes occur:

- Torch purge with argon gas
- RF power match
- Plasma ignition
- Slide valve open
- Electronics on

If the ignition process consistently fails, contact the service technician.

10.2.5.4. Allow the instrument at least 30 minutes to reach thermal equilibrium prior to starting analytical measurements. The optimization (tuning), performance testing and instrument set-up calibrations may be performed after 15 minutes. Ensure that the peristaltic pump is operated at a default analytical speed from the instrument manual.

10.2.5.5. During the initial 15 minutes, the system can be “conditioned” by aspirating the system thoroughly with 2% nitric acid + 1% HCL solution prior to continuing. (See Appendix 1 for acid preparation.)

10.2.5.6. Instrument tuning (optimization) is performed using a 20 µg/L Tune Solution, aspirated through the sample uptake tube. Optimization may not be necessary from day to day if the sample introduction system and cones have not been adjusted in any way and if the instrument fulfils the performance requirements given below. If the instrument gives performance exceeding the requirements shown below, proceed to 10.2.5.7. Otherwise, tune the instrument manually or using automated tuning while aspirating 20 µg/L Tune Solution through the sample probe, using an appropriately defined sequence is advised by the instrument manual.

The final conditions must give the following:

^{9}Be	< 500
^{115}In	< 10,000
^{208}Pb	< 10,000
^{156}CeO/^{140}Ce	<0.05

- The Collision Cell Technology (CCT) aspirates the same 20 ug/L Tune Solution listed above using a CCT defined instrument sequence. The CCT sequence uses all the same modes(lense/stage/nebulizar/ settings) as the standard tune. The CCT gaseous mixture is gradually added during the CCT tune sequence. The final CCT tune conditions must give the following:

^{115}In	> 10,000 cps
^{208}Se/Ar-Ar	< 200 cps

- If the above criteria are met, proceed to 10.2.5.7. If the above criteria are not met, do not proceed. Check that the tune solution was prepared properly and remake if necessary. If the sensitivity is below the minimum requirement, a new detector plateau may be required (refer to instrument manual), the cones may require cleaning (see Appendix 5), or the nebulizer or sample uptake lines may have become blocked or may not be properly clamped on the peristaltic pump. If the CeO/Ce ratio is >0.05, the nebulizer gas flow can be reduced and/or the sampling depth increased, obtaining a corresponding reduction in oxide formation. Recheck the above parameters after taking any remedial action.

10.2.5.7. Save the satisfactory instrument settings. Note that this is not necessary if Autotune has been used, as the instrument settings are saved automatically (unless manual adjustments have been made after autotuning).

10.2.5.8. Set-up the resolution as described in the instrument manual.

10.2.5.9. Perform a cross-calibration (and mass-calibration and detector voltage setup if required) as required by the instrument manual. Note that retuning may be necessary after performing this routine.

10.2.5.10. Aspirate Tune solution and run the tune report to confirm the mass-calibration, resolution, minimum sensitivity and maximum cerium oxide requirement and to verify instrument stability. The performance report acquires five consecutive one-minute runs and calculates the percentage relative standard deviation (RSD) of the five measurements for each isotope. The RSD of the elemental elements in the performance report must be <5%. If the performance report passes, proceed to (10.3). If the performance report fails, check:

a. Liquid uptake tubes for kinks or other damage

b. Condition and position of the peristaltic pump tubing

c. Tightness of the peristaltic pump clamp screws (these should be just tight enough to draw liquid through the tube smoothly)

d. Joints of all sample introduction components, ensuring a good seal

e. Nebulizer for blockage

f. Salt deposition on cones

- Remedy the above as necessary and repeat the test. Note that retuning may be required if any sample introduction components are adjusted or replaced.
- Resolution set-up may require adjustment if the resolution check fails. Note that the quadrupole and hexapole bias strongly influence abundance sensitivity (Pole Bias should be kept >+4V and Hexapole Bias <-3V).
- If the measured mass position for each mass in the performance report is not within ±0.1 amu of the nominal mass position, a new mass-calibration must be performed.

10.3. Sample Analysis

10.3.1. Open the last method/run sequence that contains all the saved analytical parameters. For work flow chart, refer to Appendix 7.

NOTE: The CCT Template uses both the standard and CCT modes. First the instrument analyzes in standard mode for non-CCT elements (Be, B, Ca, K, Li, Mn, Sc, Si and Ti), then it switches to CCT mode which adds the CCT gaseous mixture to analysis for the CCT elements (Al, As, B, Ba, Cd, Co, Cr, Cu, Fe, Mg, Mo, Na, Ni, P, Pb, Sb, Se, Sn, Sr, Tl, Th, U, V and Zn. Critical CCT elements are bolded). The instrument stabilizes for 20 seconds and then analysis for the CCT elements.

10.3.2. Create a run sequence or sample list that contains all required the information about calibration, QC and samples to be run. The calibration and QC concentration information should already be stored in each instrument's settings. Enter all unknown samples into the list in the appropriate order below the existing calibration and QC samples by overwriting the sample label fields. Delete any QC samples that do not apply to the required method.

10.3.3. Once all the sample information is added, check the required autosampler positions have been correctly entered. Amend as necessary. Ensure that all samples have one survey run and 3 main runs and an appropriate probe depth. Save the sequence.

10.4. Loading the Autosampler

10.4.1. Pour the required samples into pre-cleaned polypropylene test tubes. To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any possible residual contamination.

10.4.2. Pour blanks, standards and QCs into pre-cleaned polypropylene tubes. To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any possible residual contamination. Note that 2% nitric acid (7.3.1) is used as the calibration blank, ICB, and CCB.

10.4.3. For the serial dilution sample(s), dispense 2.00±0.02 mL of the original sample into a pre-cleaned 15 mL polypropylene test-tube and add 8.00±0.08 mL of 2% nitric acid (7.3.1). Mix well. This is a 5-fold dilution.

10.4.4. Place the QC and sample tubes into the appropriate position in the autosampler rack according to the sample list.

10.4.5. Place the sample probe into the autosampler arm and the internal standard probe into the internal standard solution. Run the sequence according to the steps in the instrument manual.

10.4.6. Monitor the calibration and analysis in the instrument software as the run progresses.

10.5. Post-Analysis Data Processing

10.5.1. Internal Standards

10.5.1.1. Check the internal standard recovery percentage for each internal standard isotope used for every sample. The percentage for each isotope must be within the range 30-120% for method 6020, 30–150% for methods 6020A and 6020B, and 60–125% for method 200.8.

Note: For samples from South Carolina, Internal Standard Responses must be within limits of 70-130% for samples and QC for methods 6020A and 6020B.

10.5.1.2. If above 120%, check that the other internal standard isotopes show similar deviation. If not, this may be due to the presence of the internal standard element in the sample. This is particularly common with the isotopes of Li, Sc and Y in environmental materials. If this is the case, the affected internal standard isotope may be excluded for the sample affected.

10.5.1.3. If any internal standard isotope is outside the required range (see section 10.5.1.1) and all other internal standard isotopes show similar values for that sample, the instrument may have drifted, or the sample may be producing a suppression or enhancement effect. Find the nearest blank following the sample in question and check its internal standard results. If these are similarly reduced or elevated, the instrument has drifted and the samples must be reanalyzed from the last compliant blank. If the blank does not exhibit similar drift, the sample must be producing a suppression or enhancement effect due to its matrix. In this case the sample must be reanalyzed after a five-fold (1+4) for 6020B or a ten-fold (1+9) dilution for 6020/6020A to reduce the matrix effect.

10.6. Initial Calibration

10.6.1. Open a new dataset using the date and instrument in the title. For instance the first run (A) on instrument X on January 1, 2017 would be X170101A.

10.6.2. Open the appropriate method if one already exists or create a new one for the elements to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new method.

10.6.3. See Tables 10, 11, and 12 for recommended isotopes and interference equations for commonly analyzed elements.

10.6.4. If no recommended isotopes are given for the element to be analyzed, consult a senior ICP-MS operator or appropriate reference.

10.6.5. See Table 14 for commonly used internal standards.

10.6.6. All masses which could affect data quality should be monitored to determine potential interferences either simultaneously during an analytical run or in a separate scan.

10.6.7. Internal standards are added to all standards and samples by the instrument prior to analysis.

10.6.8. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.

10.6.9. Calibration consists of a blank and a single calibration standard (see Table 2 for concentrations) in accordance with the manufacturer’s procedure. Use the average of three integrations for both calibration and sample analyses.

10.7. The order of analysis for the initial QC samples and calibration should be:

1. *Rinse*

2. *Performance Report (Tune Check)*
3. *STD1 (Blank)*
4. *STD2 (All elements except B, Mo, Sb, Si, Sn and Ti)*
5. *STD3 (B, Mo, Sb, Si, Sn and Ti)*
6. *High Linear Range Verification (Method 6020B only)*
7. *ICV (Second source, must be ± 10% of true value)*
8. *ICB*
9. *CRI / RLV/LLICV/ICVL (Reporting Limit Verification Standard)*
10. *ICSA (Interference check solution.)*
11. *ICSAB (Interference check solution, ± 20% of true value)*
12. *CCV*
13. *CCB*
14. *Prep QC such as LCS or MB, followed by samples (up to 10 runs/injections)*
15. *CCV*
16. *CCB*
17. *CRI / RLV/LLCCV/CCVL (Method 6020A only at the end of the analytical sequence)*
18. *CCV*
19. *CCB*

10.7.1. To continue the analytical run, add an additional 10 runs followed by a CCV/CCB, and repeat for up to 24 hours.

10.7.2. Analysis sequence when out-of-control QC is observed: Recalibrate and rerun all affected samples (including initial QC)

11. CALCULATIONS / DATA REDUCTION

11.1. All pertinent calculations are performed by the Mass Hunter Software. Elemental equations used to calculate results are given in Table 13.

11.2. Reporting Requirements

11.2.1. Units are ug/L or mg/L for aqueous samples and mg/kg for soil samples and ug/wipe for wipe samples.

11.2.2. If dilutions were required due to insufficient sample, interferences, or other problems, the laboratory reporting limits are multiplied by the dilution factor.

11.2.3. Document any non-standard procedures or anomalies by using the TALS NCM Module.

11.3. Data Package Requirements

11.3.1. A complete data package consists of: the daily tuning package, and all raw data.

11.3.2. All data is reviewed by a senior level laboratory analyst familiar with the technical aspects of ICP-MS and in accordance with the ICP-MS DATA REVIEW checklists.

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. Method Detection Limit Study – A Method Detection Limit (MDL) study, as described in the Detection Limit SOP, PT-QA-007, must be performed initially and whenever a significant change is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.4. Instrument Detection Limit (IDL) - IDLs are useful means to evaluate the instrument noise level and response changes over time for each element from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Section 9.3.8.

12.4.1. For all methods, IDLs must be determined for all elements. The IDL for each element must be determined for each wavelength used on each instrument. IDLs must be determined at the frequency set down by the analytical method, and any time there are any changes made to an instrument that may affect the IDL.

12.4.1.1. Each individual measurement must be performed as though it were a separate analytical sample.

12.4.1.2. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples.

12.4.1.3. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.

12.4.2. The IDLs for method 200.8 must be determined annually.

12.4.2.1. For 200.8, the IDL is determined by performing 10 replicate blank analyses and multiplying the resulting standard deviation by 3.

12.4.3. The IDLs for method 6020 must be determined quarterly.

12.4.3.1. For 6020 & 6020A, the IDLs shall be determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of the reagent blank solution with seven consecutive measurements per day.

12.4.4. The IDLs for method 6020B will be determined annually.

12.4.4.1. For 6020B, the IDLs in ug/L are calculated as the mean of the blank result plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (If the mean result is negative, zero is used for the calculation.)

12.5. Linear Range Verification (LR) - The linear range is determined semi-annually (2x/year) for each element on the standard list. **For method 6020B, the Linear Range must be verified daily with a single standard that defines the upper calibration limit.** Some regulatory programs may also require more frequent determinations. (See Table 5 for 6020B Linear Range Concentrations.)

12.5.1. To determine the linear range for methods 200.8, 6020 and 6020A, analyze 3 standards at increasing concentration up to 90% of the last concentration where the element was within 10% of true value is considered the upper linear range.

12.5.2. An alternative is to prepare a higher concentration standard and run this in the analytical run. This is required in each analytical run for method 6020B. If this standard is within 10% of the expected value this value can be used as the upper linear range. If recovery is outside 90-110%, a lower standard must be analyzed until the linear range can be verified within 10%. For method 6020B, this daily standard defines the upper linear range for the analysis.

13. POLLUTION CONTROL

13.1. It is Eurofins TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.” and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001 (the facility addendum). The following waste streams are produced when this method is carried out.

14.1.1. Acid waste consisting of sample and rinse solution. This waste is collected in waste containers identified as “Acid Waste”, Waste #33. It is neutralized to a pH between 6 and 9 and then discharged down a lab sink.

14.1.2. Expired Metals Standards. This waste is collected in waste containers identified as “Acid Waste with Metals”, Waste #6.

15 REFERENCES

14.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994

14.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, February, 2007

14.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update V, Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, October, 2012

14.5. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4, 1994

14.6. Agilent ICP-MS 7800/7900 Users' Manual

14.7. SOP PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

14.8. SOP PT-QA-006, Procurement of Standards and Materials; Labelling and Traceability

14.9. SOP PT-QA-007, Detection Limits

14.10. SOP PT-QA-016, Nonconformance & Corrective Action System

14.11. SOP PT-QA-018, Technical Data Review Requirements

14.12. SOP PT-QA-021, TestAmerica Pittsburgh QC Program

14.13. SOP PT-QA-022, Equipment Maintenance

14.14. SOP PT-QA-024, Subsampling

14.15. SOP PT-QA-031, Internal Chain of Custody

14.16. PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15. METHOD MODIFICATIONS

15.1. Use of Collision Cell Technology is an allowed method modification for SW-846 methods. Use for method 200.8 is not allowed for drinking water analysis, but may be used for waste waters.

15.2. Sample analysis using Collision Cell Technology must meet all method criteria in the same manner as analysis without it, including IDL, MDL, IDOC, Calibration and all batch QC.

16. ATTACHMENTS

Table 1 – Standard Element List and Reporting Limits

Table 2 – Composition of the CAL Standard

Table 3 – Composition of the ICV Standard

Table 4 – Composition of the CCV Standard

Table 5 – Composition of the 6020B High Linear Range Verification Standard

Table 6 – Composition of the CRI Standard

Table 7 – Composition of the Internal Standard Mix

Table 8 – Composition of the ICSA Standard

Table 9 – Composition of the ICSAB Standard

Table 10 – Common Molecular Ion Interferences in ICP-MS

Table 11 – Recommended Analytical Isotopes And Additional Masses That May Be Monitored

Table 12 – Recommended Isotopes And Additional Masses That May Be Monitored

Table 13 – Elemental Equations Used To Calculate Results

Table 14 – Internal Standards and Limitations Of Use

Table 15 - TestAmerica Pittsburgh Metals Dilution Calculation Table

Appendix 1 - Wash Solution Preparation Instructions

Appendix 2 - Daily Instrument Maintenance

Appendix 3 - Example Sample Introduction Plumbing Diagram

Appendix 4 - Procedure for Cleaning Sample Introduction Equipment and Cones

Appendix 5 - Spiking Levels

Appendix 6 - Work Flow Chart

17. REVISION HISTORY

17.1. Revision 5, 5/19/2008
17.2. Revision 6, 3/31/2009
17.3. Revision 7, 7/27/2009
17.4. Revision 8, 3/1/2011
17.5. Revision 9, 5/8/2012
17.6. Revision 10, 8/2/2013
17.7. Revision 11, 3/17/2015
17.8. Revision 12, 11/29/2016
17.9. Revision 13, 12/7/2017
17.10. Revision 14, 9/6/2019
17.11. Revision 15, 4/28/2020
17.12. Revision 16, 5/29/2020

17.13. Changes to current revision

SOP section	**Change from**	**Change to**	**Reason**
9.1 Table for CCV, 10.2.4.1 and 10.7 #14	samples	injections	Clarification
9.2.1.2		Updated the language to note that samples will be rerun along with a failing MB for the affected elements; Also added a statement that if after reanalysis MB contamination persists then the entire batch will be re-digested and reanalyzed	Clarification
9.2.2.1		Added “and all associated samples” to reanalysis/re-digestion requirement.	Clarification

TABLE 1

STANDARD ELEMENT LIST AND REPORTING LIMITS*

Element	Symbol	CAS #	Aqueous RL mg/L	Aqueous QC SPIKE mg/L	Soil/Tissue RL mg/Kg	Soil/Tissue QC SPIKE mg/kg	Wipe RL ug/wipe	Wipe QC SPIKE ug/wipe
Aluminum	Al	7429-90-5	0.03	5.0	3.0	500	1.5	250
Antimony	Sb	7440-36-0	0.002	0.25	0.2	25	0.1	12.5
Arsenic	As	7440-38-2	0.001	1.0	0.1	100	0.05	50
Barium	Ba	7440-39-3	0.010	1.0	1.0	100	0.5	50
Beryllium	Be	7440-41-7	0.001	0.50	0.1	50	0.05	25
Boron	B	7440-42-8	0.080	1.25	8.0	125	4.0	62.5
Cadmium	Cd	7440-43-9	0.001	0.50	0.1	50	0.05	25
Calcium	Ca	7440-70-2	0.500	25	50	2500	25	1250
Chromium	Cr	7440-47-3	0.002	0.50	0.2	50	0.1	25
Cobalt	Co	7440-48-4	0.0005	0.5	0.05	50	0.025	25
Copper	Cu	7440-50-8	0.002	0.50	0.2	50	0.1	25
Iron	Fe	7439-89-6	0.05	5.0	5.0	500	2.5	250
Lead	Pb	7439-92-1	0.001	0.50	0.1	50	0.05	25
Lithium	Li	7439-93-2	0.005	0.50	0.5	50	0.25	25
Magnesium	Mg	7439-95-4	0.500	25	50	2500	25	1250
Manganese	Mn	7439-96-5	0.005	0.5	0.5	50	0.25	25
Molybdenum	Mo	7439-98-7	0.005	0.5	0.5	50	0.25	25
Nickel	Ni	7440-02-0	0.001	0.5	0.1	50	0.05	25
Potassium	K	7440-09-7	0.500	25	50	2500	25	1250
Phosphorus	P	7723-14-0	0.500	1.0	50	100	25	5000
Selenium	Se	7782-49-2	0.005	1.0	0.5	100	0.25	100
Silicon	Si	7440-21-3	0.5	1.0	5.0	100	2.5	100
Silver	Ag	7440-22-4	0.001	0.25	0.1	25	0.05	12.5
Sodium	Na	7440-23-5	0.500	25	50	2500	25	1250
Strontium	Sr	7440-24-6	0.005	0.5	0.5	50	0.25	25
Tin	Sn	7440-31-5	0.005	1.0	1.0	100	0.50	50

TABLE 1

STANDARD ELEMENT LIST AND REPORTING LIMITS*

Element	Symbol	CAS #	Aqueous RL mg/L	Aqueous QC SPIKE mg/L	Soil/Tissue RL mg/Kg	Soil/Tissue QC SPIKE mg/kg	Wipe RL ug/wipe	Wipe QC SPIKE ug/wipe
Titanium	Ti	7440-03-26	0.005	0.50	0.5	50	0.25	25
Thallium	Tl	7440-28-0	0.001	1.0	0.1	100	0.05	50
Thorium	Th	7440-29-1	0.001	0.05	0.1	5	0.05	2.5
Uranium	U	7440-61-1	0.001	0.05	0.1	5	0.05	2.5
Vanadium	V	7440-62-2	0.001	0.5	0.1	50	0.05	25
Zinc	Zn	7440-66-6	0.005	0.25	0.5	25	0.25	12.5

***** *Note: These are the routine reporting limits for most sample types. Lower reporting limits may be achievable for special projects. Difficult sample matrices may cause reporting limits to be raised.*

TABLE 2

Composition of the Calibration Standards

Element	**Concentration ug/mL**	**Element**	**Concentration ug/mL**
Ag	0.200	Mo	0.40
Al	100	Na	100
As	0.40	Ni	0.40
B	1.00	P	5.0
Ba	0.40	Pb	0.40
Be	0.40	Sb	0.20
Ca	100	Se	0.40
Cd	0.40	Si	4.0
Co	0.40	Sn	0.40
Cr	0.40	Sr	0.40
Cu	0.40	Ti	0.40
Fe	100	Tl	0.20
K	100	Th	0.20
Li	0.40	U	0.20
Mg	100	V	0.40
Mn	0.40	Zn	0.20

TABLE 3

Composition of the ICV Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.08	Mo	0.08
Al	0.4	Na	40
As	0.08	Ni	0.08
B	0.40	P	2
Ba	0.08	Pb	0.08
Be	0.08	Sb	0.08
Ca	40	Se	0.08
Cd	0.08	Si	4.0
Co	0.08	Sn	0.08
Cr	0.08	Sr	0.08
Cu	0.08	Ti	0.08
Fe	20	Tl	0.08
K	40	Th	0.08
Li	0.08	U	0.08
Mg	40	V	0.08
Mn	0.4	Zn	0.08

TABLE 4
Composition of the CCV Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.1	Mo	0.2
Al	50	Na	50
As	0.2	Ni	0.2
B	0.5	P	2.5
Ba	0.2	Pb	0.2
Be	0.2	Sb	0.1
Ca	50	Se	0.2
Cd	0.2	Si	2
Co	0.2	Sn	0.2
Cr	0.2	Sr	0.2
Cu	0.2	Ti	0.2
Fe	50	Tl	0.1
K	50	Th	0.1
Li	0.2	U	0.1
Mg	50	V	0.2
Mn	0.2	Zn	0.1

TABLE 5
Composition of the 6020B High Linear Range Verification Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.200	Mo	0.200
Al	500	Na	500
As	0.200	Ni	15.0
B	0.200	P	5.0
Ba	15.0	Pb	20.0
Be	0.200	Sb	0.200
Ca	1000	Se	0.200
Cd	0.200	Si	10.0
Co	0.200	Sn	0.200
Cr	15.0	Sr	0.200
Cu	2.5	Ti	0.200
Fe	500	Tl	0.200
K	300	Th	0.200
Li	0.200	U	0.200
Mg	1000	V	0.200
Mn	20.0	Zn	20.0

TABLE 6
Composition of the CRI Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.001	Mo	0.005
Al	0.03	Na	0.5
As	0.001	Ni	0.001
B	0.08	P	0.5
Ba	0.01	Pb	0.001
Be	0.001	Sb	0.002
Ca	0.5	Se	0.005
Cd	0.001	Si	0.5
Co	0.0005	Sn	0.005
Cr	0.002	Sr	0.005
Cu	0.002	Ti	0.005
Fe	0.05	Tl	0.001
K	0.5	Th	0.001
Li	0.005	U	0.001
Mg	0.5	V	0.001
Mn	0.005	Zn	0.005

TABLE 7
Composition of the Internal Standard Mix

Element	Concentration (ug/mL)
Bi-209	0.6
Ho	0.04
In-115	0.1
Li-6	0.2
Rh	0.6
Sc	0.4
Tb	0.04
Y-89	0.1

TABLE 8
Composition of the ICSA Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Al	100	P	100
Ca	100	S	100
Fe	100	C	200
K	100	Cl^-	1000
Mg	100	Mo	2.0
Na	100	Ti	2.0

TABLE 9

Composition of the ICSAB Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.02	Na	100
Al	100	Ni	0.02
As	0.02	Pb	0.02
B	0.05	Sb	0.02
Ba	0.02	Se	0.05
Be	0.02	Si	0.50
Ca	100	Sn	0.10
Cd	0.02	Sr	0.02
Co	0.02	Ti	2.0
Cr	0.02	Tl	0.02
Cu	0.02	Th	0.02
Fe	100	U	0.02
K	100	V	0.02
Li	0.02	Zn	0.025
Mg	100.0	P	100
Mn	0.0225	S	100
Mo	2.00	C	200
		Cl-	1000

TABLE 10[1]

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

Molecular Ion	Mass	Element Interferences[2]	Molecular Ion	Mass	Element Interferences[2]
BACKGROUND MOLECULAR IONS					
NH^+	15		$^{38}ArH^+$	39	
OH^+	17		$^{40}ArH^+$	41	
OH_2^+	18		CO_2^+	44	
C_2^+	24		CO_2H^+	45	Sc
CN^+	26		ArC^+, ArO^+	52	Cr
CO^+	28		ArN^+	54	Cr
N_2^+	28		$ArNH^+$	55	Mn
N_2H^+	29		ArO^+	56	
NO^+	30		$ArOH^+$	57	
NOH^+	31		$^{40}Ar^{36}Ar^+$	76	Se
O_2^+	32		$^{40}Ar^{38}Ar^+$	78	Se
O_2H_+	33		$^{40}Ar_2^+$	80	Se
$^{36}ArH^+$	37				
MATRIX MOLECULAR IONS – Chloride					
$^{35}Cl0^+$	51	V	$^{37}Cl0H^+$	54	Cr
$^{35}Cl0H^+$	52	Cr	$^{35}Cl0^+$	51	V
$^{37}Cl0^+$	53	Cr	$^{35}Cl0H^+$	52	Cr
$Ar^{35}Cl^+$	75	As	$Ar^{37}Cl^+$	77	Se
MATRIX MOLECULAR IONS – Sulfate					
$^{32}SO^+$	48		$^{34}SOH^+$	51	V
$^{32}SOH^+$	49		SO_2^+, S_2^+	64	Zn
$^{34}SO^+$	50	V, Cr			
$Ar^{32}S^+$	72		$Ar^{34}S^+$	74	
MATRIX MOLECULAR IONS – Phosphate					
PO^+	47		PO_2^+	63	Cu
POH^+	48				
ArP^+	71				
MATRIX MOLECULAR IONS – Group I, II Metals					
$ArNa^+$	63	Cu	$ArCa^+$	80	
ArK^+	79				
MATRIX OXIDES[3]					
TiO	62-66	Ni, Cu, Zn	MoO	108-116	Cd
ZrO	106-112	Ag, Cd			

[1] From Method 200.8, Section 13.2.6

[2] Method elements or internal standards affected by the molecular ions.

[3] Oxide interferences will normally be very small and will only impact the method elements when present at

relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method element.

TABLE 11

RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES THAT MAY BE MONITORED [1]

Isotope	Element of Interest	Isotope	Element of Interest
27	Aluminum [2]	80,**78**,**82**,**76**,**77**,74	Selenium
121,123	Antimony [2]	**107**,**109**	Silver [2]
75	Arsenic [2]	**23**	Sodium [2]
138,**137**,136,**135**,134,132,130	Barium [2]	203, **205**	Thallium [2]
9	Beryllium [2]	232	Thorium
114,112,**111**,110,113,116,106,**108**	Cadmium [2]	238	Uranium
42,**43**,**44**,46,48	Calcium [2]	**51**,**50**	Vanadium [2]
52,**53**,**50**,54	Chromium [2]	**66**, **68**	Zinc [2]
59	Cobalt [2]	83	Krypton
63,**65**	Copper [2]	72	Germanium
56,**54**,**57**,58	Iron [2]	139	Lanthanum
206,207,**208**	Lead [2]	140	Cerium
24,**25**,**26**	Magnesium [2]	129	Xenon
55	Manganese [2]	**118**	Tin
98,96,92,97,94,95	Molybdenum	105	Palladium
58,**60**,62,**61**,64	Nickel [2]	47,**49**	Titanium
39	Potassium [2]	125	Tellurium
6	Lithium (internal standard)	69	Gallium
7	Lithium [2]	35,37	Chlorine

[1] *From Method 6020 CLP-M, Table 9*

[2] *Element approved for ICP-MS determination by SW846 Method 6020 CLP-M*

NOTE: Isotopes recommended for analytical determination are **bolded**.

TABLE 12

RECOMMENDED ISOTOPES AND ADDITIONAL MASSES THAT MAY BE MONITORED

Rare Earth Elements	ICPMS Preferred Mass	Elemental Equations	Additional Masses
Lanthanum	138.906		
Cerium	139.905		
Praseodymium	140.907		
Neodymium	141.908	-0.125266 * ^{140}Ce	142.910, 144.912
Samarium	151.920	-0.012780 * ^{157}Gd	144.912
Europium	152.929		
Gadolinium	157.924	-0.004016 * ^{163}Dy	156.934
Terbium	158.925		
Dysprosium	163.929	-0.047917 * ^{166}Er	
Holmium	164.930		
Erbium	165.930		
Thulium	168.934		
Ytterbium	173.939	-0.005935 * ^{178}Hf	171.937
Lutetium	174.941		

TABLE 12A			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES THAT MAY BE MONITORED			
Rare Earth Elements			
Other Elements			
Boron	11.009		
Calcium	43.956		
Cesium	132.905		
Galium	68.926		
Germanium	71.922		
Gold	196.967		
Hafnium	177.944		176.944
Holmium	164.930		
Iridium	192.963		
Lithium	7.016		
Tungsten	183.951	-0001242* ^{189}Os	
Uranium	238.050		
Yttrium	88.905		
Zirconium	238.050		
Niobium	92.906		
Palladium	104.905		
Phosphorus	30.994		
Platinum	194.965		
Rhenium	186.965	-0.099379 * ^{189}Os	
Rhodium	102.905		
Rubidium	84.912		
Ruthenium	101.904	-0.045678 * ^{105}Pd	
Scandium	44.956		
Strontium	87.906		
Tantalum	180.948		
Tellurium	127.905	-0.072348 * ^{129}Xe	
Thorium	232.03		

TABLE 13
ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS

Element	Elemental Equation	Note
Al	$(1.000)\ (^{27}C)$	
Sb	$(1.000)\ (^{121}C)$	
As	$(1.000)\ (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]$	Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
Ba	$(1.000)\ (^{137}C)$	
Be	$(1.000)\ (^{9}C)$	
Cd	$(1.000)\ (^{111}C) - (1.073)\ [(^{108}C) - (0.712)\ (^{106}C)]$	Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.
Cr	$(1.000)\ (^{52}C)$	In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
Co	$(1.000)\ (^{59}C)$	
Cu	$(1.000)\ (^{63}C)$	
Pb	$(1.000)\ (^{206}C) + (1.000)\ (^{207}C) + (1.000)\ (^{208}C)$	Allowance for isotopic variability of lead isotopes.
Li	$(1.000)\ (Li^{6}) - (Li^{7})\ (0.037)$	
Mn	$(1.000)\ (^{55}C)$	
Mo	$(1.000)\ (^{98}C) - (0.146)\ (^{99}C)$	Isobaric elemental correction for ruthenium.
Ni	$(1.000)\ (^{60}C)$	
Se	$(1.000)\ (^{82}C)$	Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
Ag	$(1.000)\ (^{107}C)$	
Tl	$(1.000)\ (^{205}C)$	
Th	$(1.000)\ (^{232}C)$	
U	$(1.000)\ (^{238}C)$	
V	$(1.000)\ (^{51}C) - (3.127)\ [(^{53}C) - (0.113)\ (^{52}C)]$	Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ratio may be determined from the reagent blank.
Zn	$(1.000)\ (^{66}C)$	

TABLE 13 ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS		
Internal Standards		
Bi	(1.000) (^{209}C)	
In	(1.000) (^{115}C) -(0.0149) (^{118}C)	Isobaric elemental correction for tin.
Ge	(1.000) (^{72}C)	
Sc	(1.000) (^{45}C)	
Tb	(1.000) (^{159}C)	
Tm	(1.000) (^{169}C)	
Y	(1.000) (^{89}C)	

* Method elements or internal standards affected by the molecular ions. C = Calibration blank subtracted counts at specified mass.

TABLE 14

INTERNAL STANDARDS AND LIMITATIONS OF USE

Internal Standard	Mass	Possible Limitation
Lithium	6	a
Scandium	45	Polyatomic Ion Interference
Germanium	72	
Yttrium	89	a, b
Rhodium	103	
Indium	115	Isobaric Interference by Sn
Terbium	159	
Holmium	165	
Thulium	169	
Lutetium	175	
Bismuth	209	a

a May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

Table 15 - TestAmerica Pittsburgh Metals Dilution Calculation Table

DILUTION REQUIRED	Dilution Formula
2	5 mL sample : 5 mL Matrix Blank
5	2 mL sample : 8 mL Matrix Blank
10	1 mL sample : 9 mL Matrix Blank
25	0.4 mL sample : 9.6 mL Matrix Blank
50	0.2 mL sample : 9.8 mL Matrix Blank
100	0.1 mL sample : 9.9 mL Matrix Blank

Appendices

Appendix 1

Wash Solution Preparation Instructions (2% Nitric Acid (v/v))

A large volume of this solution is required for supply to the autosampler rinse station in order to wash the probe between samples. These instructions detail the preparation procedure for 2.5 L of this solution that is normally sufficient for one day of analytical use. The procedure may be scaled up or down as required.

1) Into a clean 2.5 L container add 500±450 mL of laboratory water (7.1)
2) Add 50±10 mL of concentrated nitric acid (7.3)
3) Make to 2.50±0.25 L with laboratory water (7.2)
4) Mix well

Notes:

If preparing larger quantities simply scale-up quantities proportionally.

If analyzing for Ag, add hydrochloric acid at 1% by adding 50±10 mL of concentrated hydrochloric acid (6.1.2) after step 2.

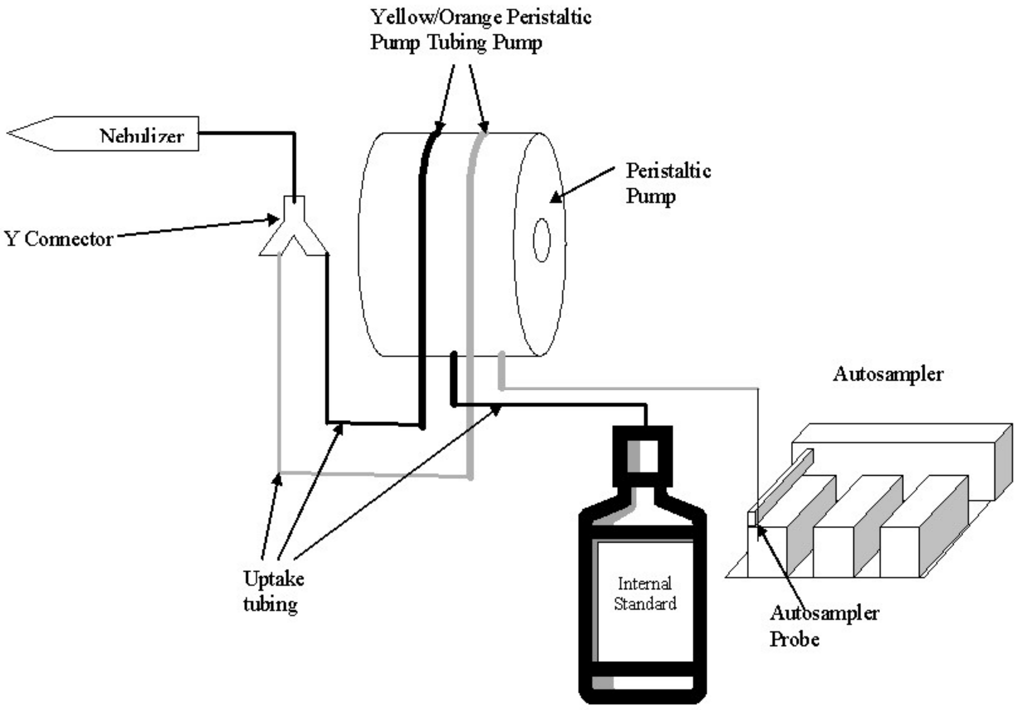
Appendix 2

Daily Instrument Maintenance

1) Wipe all instrument, autosampler and surrounding bench surfaces with a damp wipe – continual cleanliness is important for the minimization of contamination
2) Check Wash Solution volume and remake if necessary (see Appendix 2)
3) Empty Waste Vessel according to laboratory disposal policy
4) Check the condition of all peristaltic pump tubes and replace if required (it is recommended to replace these daily although this may not be necessary with lower sample loads)
5) Check condition of sample introduction system and cones and clean and/or replace as necessary (see Appendix 5)
6) Ensure instrument fume-extraction system is operational

Appendix 3

Example Sample Introduction Plumbing Diagram



Appendix 4

Procedure for Cleaning Sample Introduction Equipment and Cones

1) Ensure that the instrument is in the *vacuum* or *shutdown* state (i.e. the plasma is OFF and the slide valve is SHUT)
2) Dismantle the sample introduction system as follows:
a) Remove the gas connection from the nebulizer
b) Remove the sample input plug from the nebulizer
c) Remove the metal clip on the spray chamber to elbow joint
d) Remove the drain plug from the spray chamber
e) Slide the spray chamber and nebulizer away from the elbow
f) Carefully slide the nebulizer out of the spray chamber and set both pieces aside in a safe place
g) Open the torch box and the internal Faraday cage
h) Pull the gas connections away from the torch
i) Undo the torch catch
j) Remove the metal clip on the elbow to torch joint
k) Carefully remove the torch from the load coil and set aside in a safe place
l) Remove the elbow by sliding it out of the torch box bulkhead toward spray chamber end
m) Slide the torch box away from the mass spectrometer to reveal the interface
n) Use the flat metal cone tool to undo the locking ring over the sample cone
o) Carefully remove the sample cone and set aside in a safe place
p) Carefully unscrew and remove the skimmer cone from the interface using the cylindrical aluminium tool and set aside in a safe place
3) Clean the cones as follows.
a) Carefully place the cones into a large beaker and fill with sufficient deionized water.
b) Place the beaker in an ultrasonic bath for about 10 minutes or until surface deposition has been removed
c) Carefully remove the cones from the solution and rinse thoroughly with deionised water
d) Allow the cones to air-dry prior to refitting
4) Clean the sample introduction equipment as follows.
e) Carefully place the glass sample introduction components into a large beaker and fill with sufficient 10% nitric acid to cover all components
f) Place in an ultrasonic bath for 20 minutes

g) Carefully remove the glass components and rinse thoroughly with deionised water

h) Allow to air-dry prior to refitting

5) Reassemble the components in the reverse order to disassembly

Note: Occasionally, glass sample introduction components crack when the ultrasonic cleaning procedure is used. To avoid this, the components may be soaked in acid, as above, for 12 hours, without ultrasonic treatment.

Appendix 5

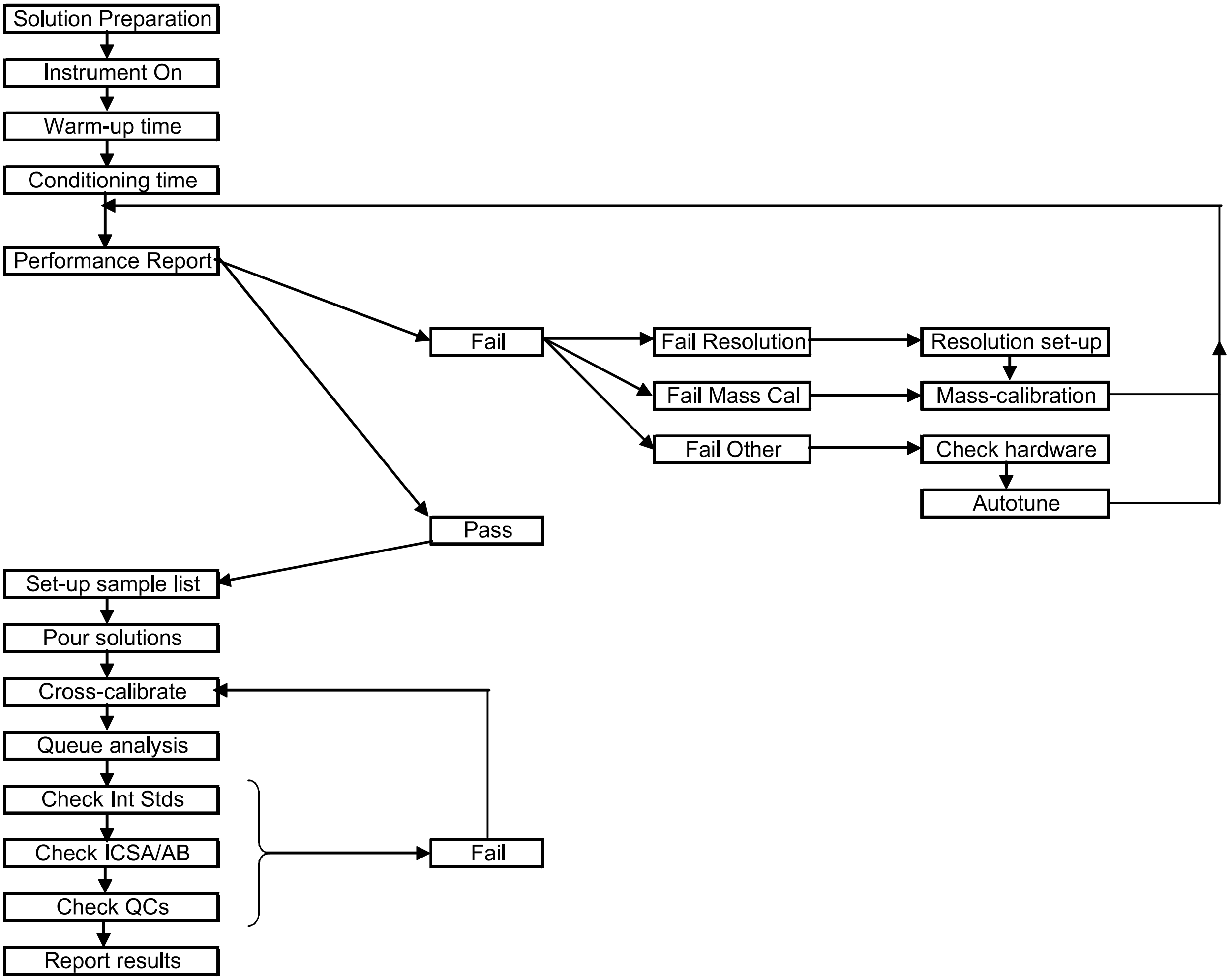
Spiking Levels

(Concentration in Final Solution Based on Instructions Within this Document)

Element	Spike Value (µg/L)	Element	Spike Value (µg/L)
Ag	250	Mo	500
Al	5000	Na	25000
As	1000	Ni	500
B	1250	P	1000
Ba	1000	Pb	500
Be	500	Sb	250
Ca	25000	Se	1000
Cd	500	Si	1000
Co	500	Sn	1000
Cr	500	Sr	500
Cu	500	Ti	500
Fe	5000	Tl	1000
K	25000	Th	50
Li	500	U	50
Mg	25000	V	500
Mn	500	Zn	250

Appendix 6

Work Flow-Chart



Appendix C
Laboratory Certification

COMMONWEALTH OF VIRGINIA
DEPARTMENT OF GENERAL SERVICES
DIVISION OF CONSOLIDATED LABORATORY SERVICES

Certifies that

VA Laboratory ID#: 460189
Eurofins Pittsburgh

301 Alpha Drive
Pittsburgh, PA 15238

Owner: EUROFINS ENVIRONMENT TESTING NORTHEAST, LLC ("EETNE")
Operator: EUROFINS PITTSBURGH
Responsible Official: DEBORAH LOWE

Having met the requirements of 1 VAC 30-46 and
having been found compliant with the 2009 TNI Standard approved by The NELAC Institute
is hereby approved as an

Accredited Environmental Laboratory

As more fully described in the attached Scope of Accreditation

Effective Date: **January 1, 2022**
Expiration Date: **September 14, 2022**
Certificate # 11661

Denise M. Toney

Denise M. Toney, Ph.D., HCLD
DGS Deputy Director for Laboratories

Continued accreditation status depends on successful ongoing participation in the program.
Certificate to be conspicuously displayed at the laboratory.
Not valid unless accompanied by a valid Virginia Environmental Laboratory Accreditation Program (VELAP) Scope of Accreditation.
Customers are urged to verify the laboratory's current accreditation status.

Certificate Not Transferable

Surrender Upon Revocation

Commonwealth of Virginia

Department of General Services
Division of Consolidated Laboratory Services

Scope of Accreditation

VELAP Certificate No.: 11661

Eurofins Pittsburgh
301 Alpha Drive
Pittsburgh, PA 15238

Virginia Laboratory ID: 460189
Effective Date: January 01, 2022
Expiration Date: September 14, 2022

NON-POTABLE WATER

METHOD	ANALYTE	PRIMARY	METHOD	ANALYTE	PRIMARY
EPA 6010 C	TIN	PA	EPA 6010 C	TITANIUM	PA
EPA 6010 C	VANADIUM	PA	EPA 6010 C	ZINC	PA
EPA 6010 D	ALUMINUM	PA	EPA 6010 D	ANTIMONY	PA
EPA 6010 D	ARSENIC	PA	EPA 6010 D	BARIUM	PA
EPA 6010 D	BERYLLIUM	PA	EPA 6010 D	BORON	PA
EPA 6010 D	CADMIUM	PA	EPA 6010 D	CALCIUM	PA
EPA 6010 D	CHROMIUM	PA	EPA 6010 D	COBALT	PA
EPA 6010 D	COPPER	PA	EPA 6010 D	IRON	PA
EPA 6010 D	LEAD	PA	EPA 6010 D	LITHIUM	PA
EPA 6010 D	MAGNESIUM	PA	EPA 6010 D	MANGANESE	PA
EPA 6010 D	MOLYBDENUM	PA	EPA 6010 D	NICKEL	PA
EPA 6010 D	POTASSIUM	PA	EPA 6010 D	SELENIUM	PA
EPA 6010 D	SILICA AS SIO2	PA	EPA 6010 D	SILVER	PA
EPA 6010 D	SODIUM	PA	EPA 6010 D	STRONTIUM	PA
EPA 6010 D	THALLIUM	PA	EPA 6010 D	TIN	PA
EPA 6010 D	TITANIUM	PA	EPA 6010 D	VANADIUM	PA
EPA 6010 D	ZINC	PA	EPA 6020 A	ALUMINUM	PA
EPA 6020 A	ANTIMONY	PA	EPA 6020 A	ARSENIC	PA
EPA 6020 A	BARIUM	PA	EPA 6020 A	BERYLLIUM	PA
EPA 6020 A	CADMIUM	PA	EPA 6020 A	CALCIUM	PA
EPA 6020 A	CHROMIUM	PA	EPA 6020 A	COBALT	PA
EPA 6020 A	COPPER	PA	EPA 6020 A	IRON	PA
EPA 6020 A	LEAD	PA	EPA 6020 A	MAGNESIUM	PA
EPA 6020 A	MANGANESE	PA	EPA 6020 A	NICKEL	PA
EPA 6020 A	POTASSIUM	PA	EPA 6020 A	SELENIUM	PA
EPA 6020 A	SILVER	PA	EPA 6020 A	SODIUM	PA
EPA 6020 A	THALLIUM	PA	EPA 6020 A	VANADIUM	PA
EPA 6020 A	ZINC	PA	EPA 6020 A - EXTENDED	BORON	PA
EPA 6020 A - EXTENDED	MOLYBDENUM	PA	EPA 6020 A - EXTENDED	STRONTIUM	PA
EPA 6020 A - EXTENDED	TIN	PA	EPA 6020 A - EXTENDED	TITANIUM	PA
EPA 6020 B	ALUMINUM	PA	EPA 6020 B	ANTIMONY	PA
EPA 6020 B	ARSENIC	PA	EPA 6020 B	BARIUM	PA
EPA 6020 B	BERYLLIUM	PA	EPA 6020 B	CADMIUM	PA
EPA 6020 B	CALCIUM	PA	EPA 6020 B	CHROMIUM	PA
EPA 6020 B	COBALT	PA	EPA 6020 B	COPPER	PA
EPA 6020 B	IRON	PA	EPA 6020 B	LEAD	PA
EPA 6020 B	MAGNESIUM	PA	EPA 6020 B	MANGANESE	PA
EPA 6020 B	MOLYBDENUM	PA	EPA 6020 B	NICKEL	PA
EPA 6020 B	POTASSIUM	PA	EPA 6020 B	SELENIUM	PA