

## Final Sampling and Analysis Plan

### Upper Current and Big Spring Small Arms Firing Ranges (SAFRs) Engineering Evaluation/Cost Analysis Ozark National Scenic Riverways (OZAR)

Prepared by

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## List of Abbreviations and Acronyms

AI	Applied Intellect, LLC
APPL	Agriculture and Priority Pollutant Laboratory
ARAR	Applicable or Relevant and Appropriate Requirement
bgs	Below ground surface
BLM	Bureau of Land Management
CAS	Chemical Abstract Service
CCV	Continuing Calibration Verification
CEC	Cation Exchange Capacity
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CIP	Community Involvement Plan
cm	centimeter
CO	Contracting Officer
COC	Chain of Custody
COPC	Constituents of Potential Concern
COPEC	Constituents of Potential Ecological Concern
COR	Contracting Officer's representative
CSEM	Conceptual Site Exposure Model
CY	Cubic Yard
DL	Detection Limit
DQA	Data Quality Assessment
DQI	Data Quality Indicator
DQO	Data Quality Objective
DU	Decision unit
EDD	Electronic data deliverable
EDL	Environmental and Disposal Liabilities
EE/CA	Engineering Evaluation/Cost Analysis
ELAP	Environmental Laboratory Accreditation Program
ESL	Environmental screening level
ESV	National Park Service Ecological Screening Value
FSP	Field Sampling Plan



GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
ID	Identifier
IDW	Investigative-derived waste
IDQTF	Intergovernmental Data Quality Task Force
ICV	Initial Calibration Verification
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology Regulatory Council
LCS	Laboratory Control Sample
LIMS	laboratory information management system
LOD	Limit of Detection
LOQ	Limit of Quantitation
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
ml	Milliliter
MS/MSD	matrix spike/matrix spike duplicate
MOPC	Medium of potential concern
NCP	National Oil and Hazardous Substances Pollution Contingency Plan (AKA, National Contingency Plan)
NELAP	National Environmental Laboratory Accreditation Program
NPS	National Park Service
NTCR	Non-Time Critical Removal Action
NWI	National Wetlands Inventory
OSHA	Occupational Safety and Health Act
OZAR	Ozark National Scenic Riverway
P&I	Plants and Invertebrates
PA	Preliminary Assessment
pH	Hydrogen potential
PM	Project Manager
PPE	Personal protective equipment



PQL	Practical quantitation limit
PRG	Preliminary Removal Goal
PSL	Project Screening level
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
RME	reasonable maximum exposure
RPD	Relative percent difference
RSL	Regional Screening Level
SAFR	Small Arms Firing Range
SAP	Sampling and Analysis Plan
SI	Site Inspection
SIM	Selective Ion Monitoring
SLRA	screening level risk assessment
SOP	Standard Operating Procedure
SS	Surface Soil
SSHP	Site Health and Safety Plan
SU	Sampling Unit
SUB	Surface and Shallow Subsurface Soil
TAL	Target Analyte List metals
TCLP	Toxic Characteristic Leaching Procedure
TOC	Total Organic Carbon
USC	United States Code
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UTM	Universal Transverse Mercator
WPA	Federal Works Progress Administration





## 1. INTRODUCTION

This document serves as the Sampling and Analysis Plan (SAP) for two Engineering Evaluation/Cost Analysis (EE/CAs) at the Upper Current Small Arms Firing Range (SAFR), Shannon County, Missouri (MO); and the Big Springs SAFR, Carter County, MO (the Sites). Both Sites are located within the Ozark National Scenic Riverways (OZAR) as shown in Figure 1-1 and Figure 1-2, respectively. The purpose of this SAP is to define:

- The purpose of this study;
- The use for the data generated;
- The quality of data needed to accomplish the goals of this study; and
- The data collection method.

### 1.1 CERCLA and National Park Service (NPS) Authority

This SAP was generated in accordance with NPS Sampling and Analysis Plan Template (NPS, 2020), which was developed in accordance with, or supersedes Environmental Protection Agency's (USEPA) guidance documents, including USEPA's Guidance on Systematic Planning Using the Data Quality Objectives Process (USEPA, 2006), Guidance for Quality Assurance Project Plans (USEPA 2002), EPA Requirements for Quality Assurance Project Plans (USEPA, 2001), and the Intergovernmental Data Quality Task Force's (IDQTF) Uniform Federal Policy for Quality Assurance Project Plans (IDQTF, 2005). The National Park Service (NPS) is authorized under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. §§ 9601 et seq., to respond as the Lead Agency to a release or threatened release of hazardous substances and/or a release or threatened release of any pollutant or contaminant that may present an imminent and substantial danger to public health or welfare on NPS land.

CERCLA's implementing regulations, codified in the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 CFR Part 300, establishes the framework for responding to such releases and threatened releases. The NCP prescribes two similar processes for responding to releases: removal actions and remedial actions (See NCP Sections 300.400 through 300.440). If environmental samples are to be collected under either process, a SAP is required (See NCP Sections 300.415 and 300.430). The SAP is comprised of two parts: a Field Sampling Plan (FSP) and a Quality Assurance Project Plan (QAPP). The FSP describes the number, types, and locations of samples as well as the types of analyses that will be conducted on the samples. The QAPP describes the project's policy, organization, and functional activities as well as the data quality objectives (DQOs) and measures necessary to achieve the goals of the study.

In addition, the NPS has several regulations that apply to the release of hazardous substances on NPS land including the NPS Organic Act of 1916 (16 USC §1, et seq. 36 CFR Part 1), which requires that the NPS manage parks to conserve the scenery, natural and historic objects, and wildlife and to provide for their enjoyment by such means as will leave them unimpaired for the enjoyment of future generations. Therefore, whether the Site poses risks to the interaction of organisms and the environment is especially relevant to the NPS responsibility to protect park resources.



To the extent practicable, all work will be conducted in accordance with the USEPA guidance, Green Remediation: Incorporating Sustainable Environmental Practices into Remediation of Contaminated Sites (USEPA, 2008).

The results of a Preliminary Assessment (PA) conducted by NPS contractor (Terracon 2006)) at the Sites identified areas of concern requiring further evaluation. Therefore, NPS has determined that a non-time critical removal action may be appropriate to address the release or threatened release of hazardous substances at the Site and an EE/CA must be completed in accordance with Section 300.415(b)(4) of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 CFR Part 300.

## **1.2 Purpose of Field Sampling**

The purpose for conducting the EE/CA field investigations are to characterize lead in soil at the Upper Current and Big Spring SAFRs to:

- Determine risks to human health of NPS personnel (Park workers) and Park visitors;
- Determine risk to ecological receptors; and
- If risks are unacceptable, conduct an alternatives screening analysis to identify cleanup remedies for each Site.

The purpose of field sampling is to determine the nature and extent of potential environmental impacts to the Site from prior small arms firing range activities. The NPS will use data collected during this field investigation to support potential response actions that may be undertaken by the NPS or other parties. Data generated from these sampling events will be used in accordance with the provisions outlined in the DQOs. The following types of data will be collected during the field investigation:

Incremental sampling methodology (ISM) following Interstate Technology and Regulatory Council (ITRC) guidelines and discrete surface and shallow subsurface soil samples for analysis of lead;

ISM and discrete surface and subsurface soil samples for analysis of characteristics important in quantifying the toxicity of metal contaminants to human and ecological receptors, namely pH;

Documentation of field conditions, including visual documentation of firing range soil particle sizes as related to bioavailability and migration potential.

NPS will use the data obtained from these investigations in accordance with the provisions outlined in the DQOs detailed in Section 4. Once the nature and extent of contamination is determined and both human health and ecological risk assessments have been completed, the alternatives for removal action, if needed, can be evaluated. The preferred alternative will be selected by NPS in the Final EE/CA to support the non-time critical removal action (NTCRA).



### 1.3 Site Locations

Figure 1-1 is the Sites Location Map for Upper Current SAFR and Big Spring SAFR. Both Sites are located in southeast Missouri, along the OZAR Notional Scenic Riverways, surrounded by the Mark Twain National Forest as shown in Figure 1-2.

#### 1.3.1 Upper Current SAFR

The Upper Current SAFR Site is located on the eastern bank of the Current River in proximity to Welch Landing. The Department of Interior (DOI) Environmental Disposal and Liability (EDL) database identifies the Site as Welch Active Firing Range, with an EDL latitude and longitude of 37.39642 North, -91.57174 East. The Site is located approximately 0.7 miles east of the Current River and 0.3 miles west of State Highway K, as shown on Figure 1-3 from the United States Geological Survey (USGS) Cedar Grove Topographic Map, dated 2017, scale 1:24,000. The site is located in the Fifth Principal Meridian, Township 31 North, Range 6 West, southwest quadrant of Section 11 at an elevation of approximately 1,120 feet above mean sea level (ft, amsl). As shown in Figure 1-4, the Site is accessible via unpaved access road running west off State Highway K at latitude 37.3984 North, -90.43326 East. The Upper Current SAFR is located in a proposed Wilderness Area, and Wilderness rules are mandated for work associated with this project. This range was active between mid-1970 and 2015. The study area is densely wooded area and is described in the EDL data base as “This range has 4 lanes, is a mowed grass area approximately 70' X 250' and is surrounded by timber.” Figure 1-5 identifies the Site Features.

#### 1.3.2 Big Spring SAFR

The Big Spring SAFR Site is located approximately 4 miles south of South Van Buren and 2 miles west of Long Bay Hollow portion of the Current River and 0.25 miles east of State Highway Z as shown on Figure 1-6 from the Big Spring, MO, 1:24,000 quad, 2017, USGS topographic map. The DOI EDL database identifies the Site as Big Spring Active Shooting Range, with an EDL latitude and longitude of 36.926367 North, -90.983967 East. The Site is located in the Fifth Principal Meridian, Township 26 North, Range 1 East, southwest quadrant of Section 8 at an elevation of approximately 500 ft, amsl. As shown in Figure 1-7, the Site is accessible via unpaved access road running west off State Highway Z at latitude 36.927011 North, -89.020216 East. The Big Spring SAFR is located in a proposed Wilderness Area, and Wilderness rules are mandated for work associated with this project. The study area is densely wooded area and is described in the EDL data base as “This range was active between mid-1970s and 2015. This range has five lanes, is a mowed grass area approximately 55 yds X 25 yds and is surrounded by timber.” Figure 1-8 identifies the Site Features.

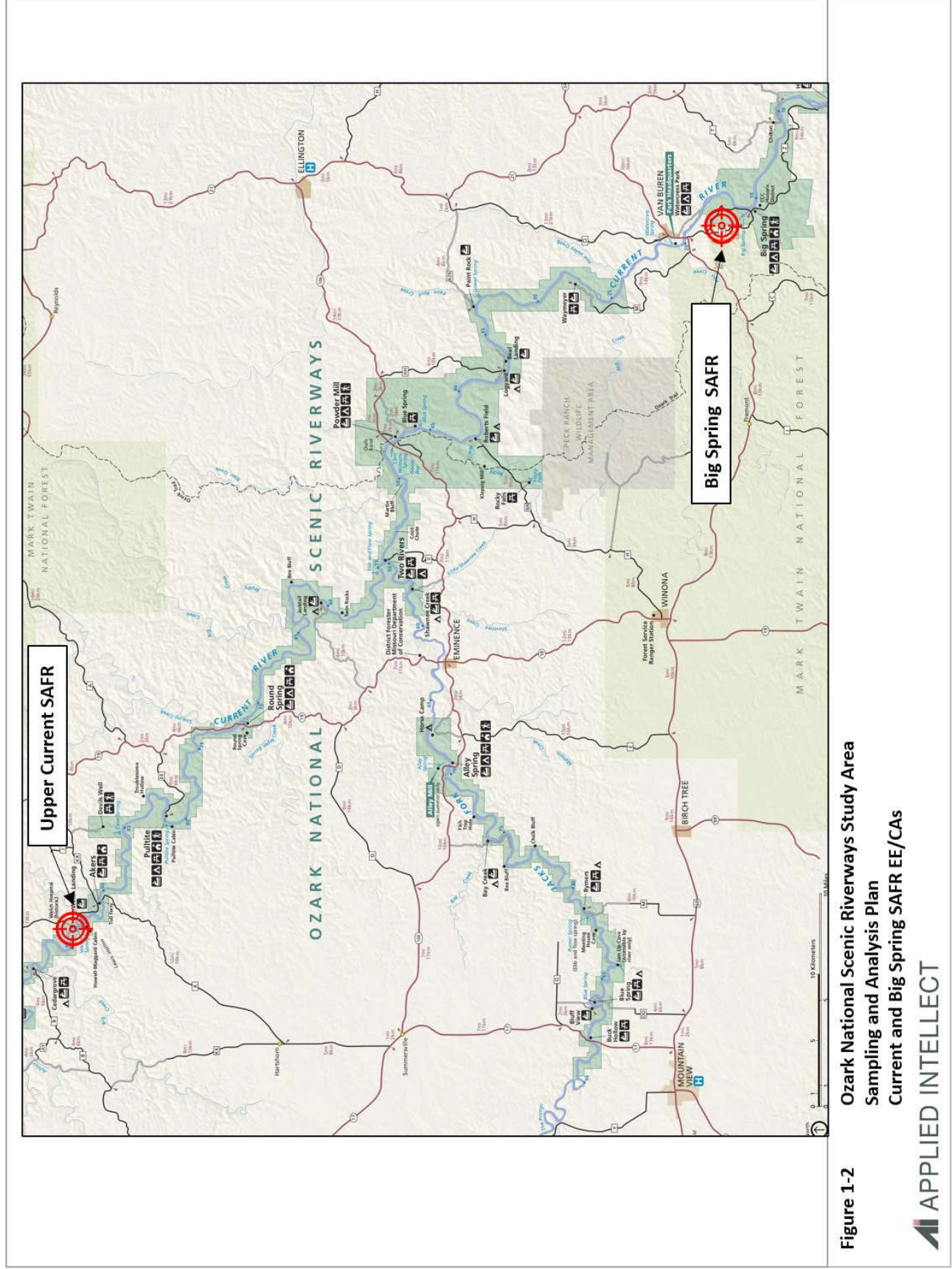




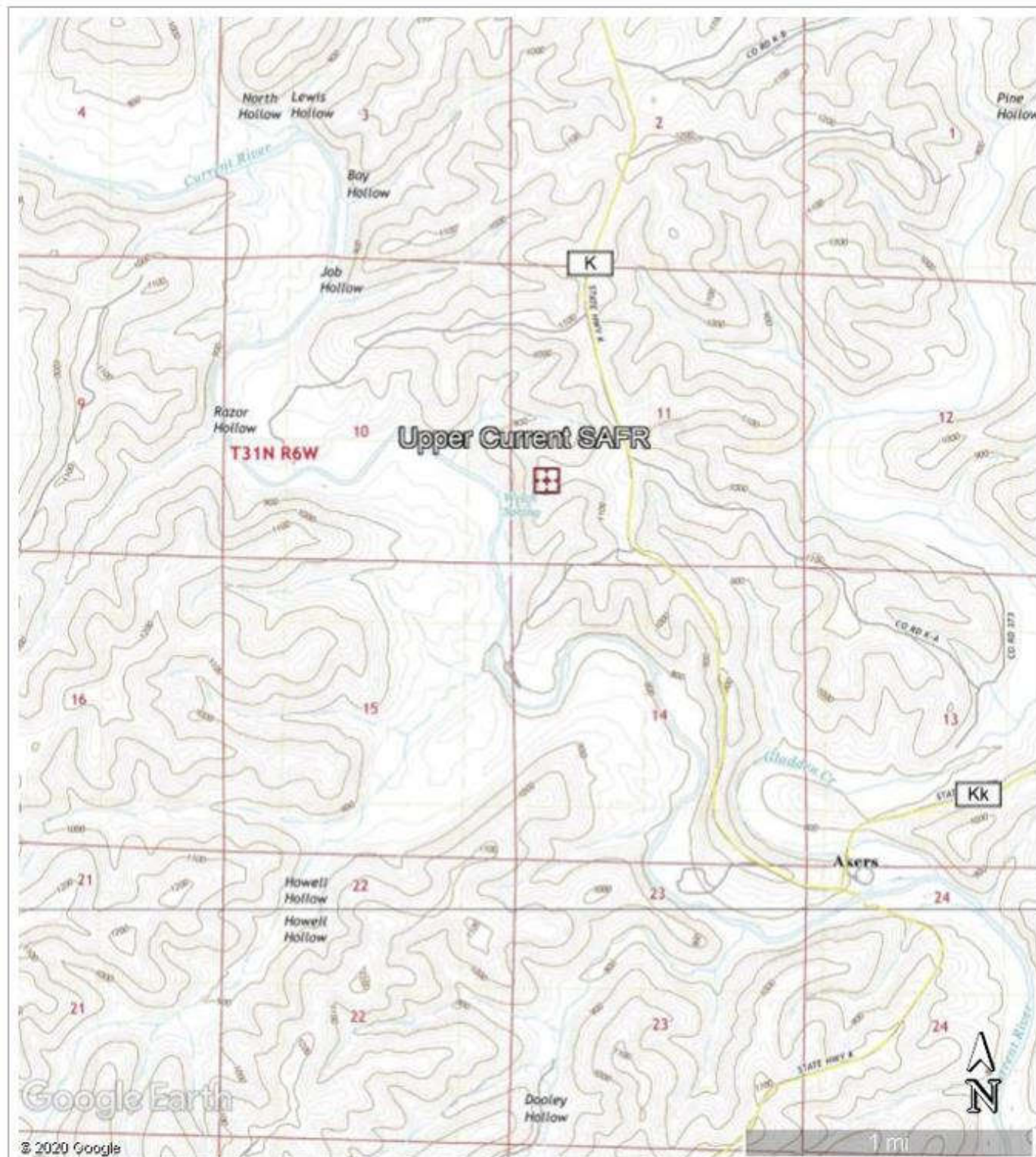
Figure 1-1 Current SAFR and Big Spring SAFR Location Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs

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**Figure 1-3** Upper Current SAFR Topographic Features Map  
National Historic Preservation Act Section 106 Compliance  
Current and Big Spring SAFR EE/CAs





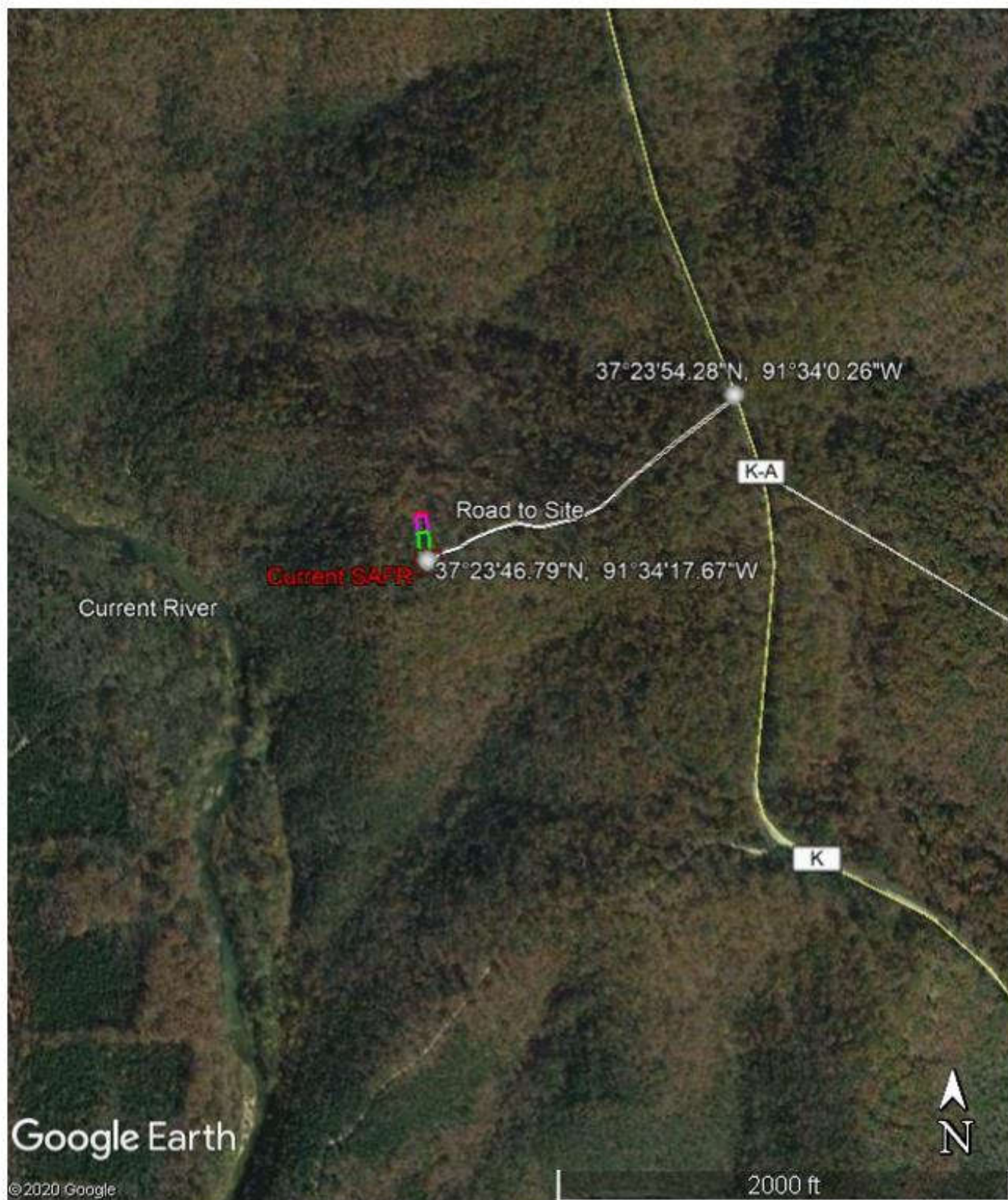


Figure 1-4 Upper Current SAFR Study Area Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs







Figure 1-5

Upper Current SAFR Features Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs





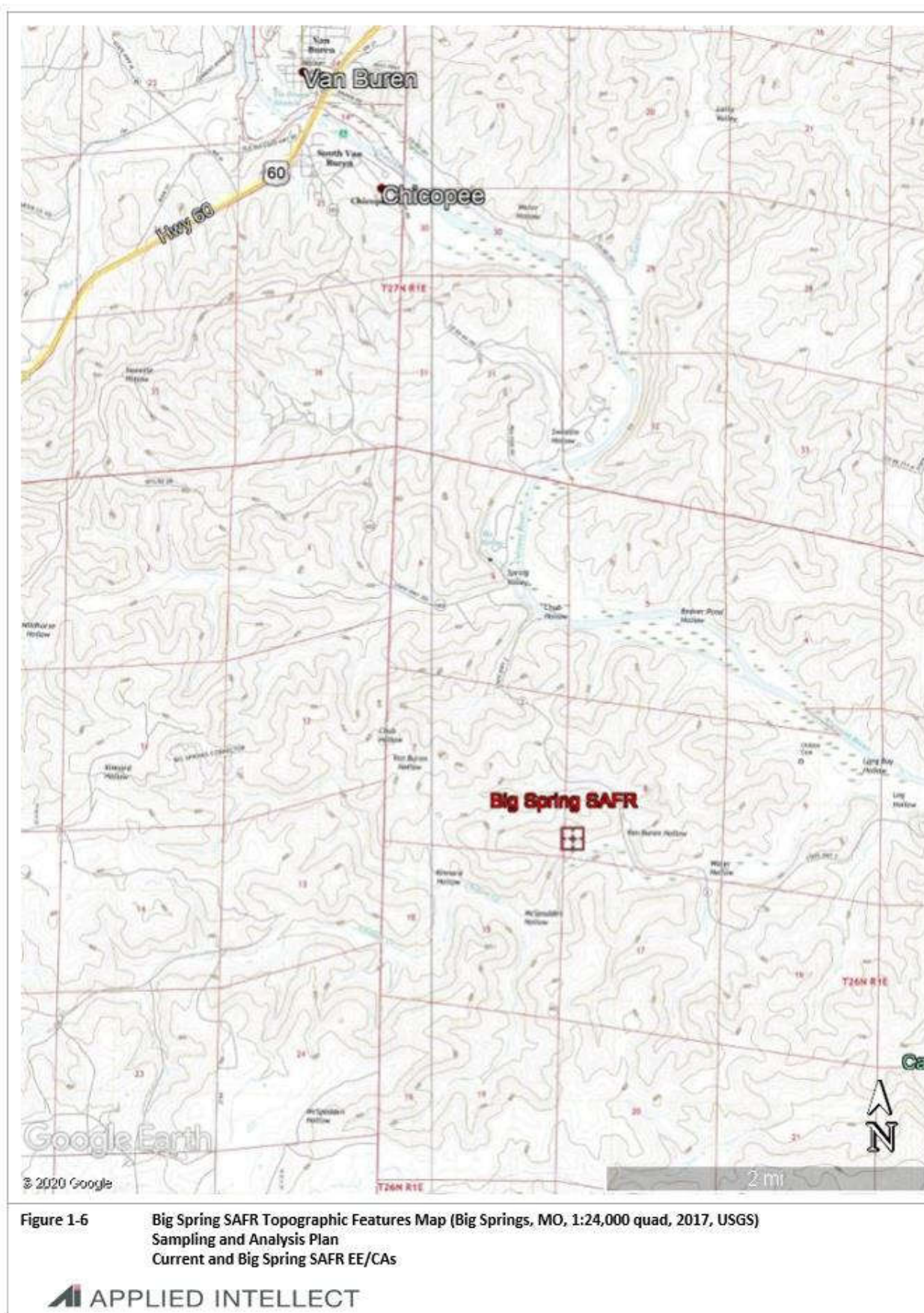










Figure 1-8      Big Springs SAFR Features Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs





## 2. SITE DESCRIPTION, PREVIOUS INVESTIGATIONS, AND CONCEPTUAL SITE MODEL

Section 2.1 describes the site features, the operational histories, waste characteristics, geology and hydrogeology, climate, and sensitive environments. Section 2.2 summarizes the previous investigations and data usability and introduces the Conceptual Site Model.

### 2.1 Key Site Features

This section provides a physical and operational description of the Site as well as information regarding locations where waste storage, handling, disposal and deposition may have occurred.

### 2.2 Site Description

Two small arms firing ranges (SAFRs) are located in the Ozark National Scenic Riverways and proposed wilderness area. The firing ranges are as follows:

- Upper Current Firing Range near Akers, Missouri; and
- Big Spring Firing Range near Big Spring, Missouri.

Information including range history, use, and layout was gathered by Terracon (Terracon 2006). The firing ranges were used for pistol and rifle range practice and training. Ammunition used at the ranges consisted of .40 and .45 caliber, 9 mm pistol rounds, .223 caliber M-16 high-powered rifle rounds, and 12 gauge shot gun ammunition including one-ounce slugs and buckshot.

Lead bullets and fragments were observed at the ranges during the September 5 site visit. No known lead reclamation efforts have been conducted at the sites.

#### 2.2.1 Upper Current SAFR

The Upper Current Firing Range site is an active 1/3-acre small-arms firing range owned by the National Park Service and located in the Ozark National Waterways and proposed wilderness area near Akers, Missouri. The site's location is depicted in Figures 1, 2, and 3 (Appendix A). The layout of the site is depicted in Figure 1-5.

The small arms firing range includes a firing line marked by wood posts, a target line marked by four plywood targets (approximately two feet wide by three feet tall) mounted on wooden posts approximately three feet off the ground, and an earthen backstop behind the targets. The subject site is a rectangular-shaped property that is approximately 70 feet by 250 feet. The area surrounding the range is heavily wooded with some small hills. The Current River is located approximately 400 yards west of the firing range.

#### 2.2.2 Big Springs SAFR

The Big Spring Firing Range site is an active 1/3-acre small-arms firing range owned by the National Park Service and located in the Ozark National Riverways and proposed wilderness area near Big Spring,





Missouri. The site's location is depicted in Figures 1, 8, and 9 (Appendix A). The layout of the site is depicted in Figure 1-9

The small arms firing range includes three firing lines marked by wood posts, a target line marked by wooden posts, and a storage building (approximately 30 feet by 50 feet) on the southeast side. There was no distinct backstop berm to stop firearms projectiles after passing through the target line. The subject site is a rectangular-shaped property that is approximately 80 feet by 200 feet. The area surrounding the range is heavily wooded with some small hills.

## **2.3 Operational History**

### **2.3.1 Upper Current SAFR**

The small arms firing range has been routinely used since approximately the 1970s and was used by approximately five park service personnel twice a year for training, practice, and qualification through 2013. Individuals who used the range included park service personnel and the Missouri Conservation Department personnel. The SAFR has been inactive since approximately 2013.

### **2.3.2 Big Springs SAFR**

The small arms firing range has been routinely used since approximately the 1970s and was used by approximately six law enforcement personnel twice a year for training, practice, and qualification. Non-park personnel who use the range were primarily Carter County Sheriffs. The SAFR has been inactive since approximately 2013.

## **2.4 Waste Characteristics**

### **2.4.1 Upper Current SAFR**

According to Terracon (2006), spent lead bullets, bullet fragments, or lead shot were observed in 9 of the 24 samples collected. Seventeen samples were analyzed for total lead. The concentration of lead in samples ranged from 23.3 to 23,600 mg/kg. The highest concentration of lead in a sample where no bullets or fragments were observed at 6 to 12 inches below ground and was 1,410 mg/kg total lead.

### **2.4.2 Big Springs SAFR**

According to Terracon (2006), spent lead bullets, bullet fragments, or lead shot were observed in eight of the 23 samples collected. Seventeen samples were analyzed for total lead. The concentration of lead in samples ranged from 11 to 7,360 mg/kg total lead. The highest concentration of lead in a sample where no bullets or fragments were observed was 786 mg/kg total lead.

## **2.5 Local Geologic and Hydrogeologic Setting**

As described in the Foundation Document, Ozark National Scenic Riverways (NPS 2016), OZAR includes portions of the Current and Jacks Fork Rivers, providing 134 miles of clear, free-flowing, spring-fed waterways. Much of the area is underlain by dolomite that has given rise to numerous sinkholes,





caves, and springs representative of classical karst topography. Up to 90% of the combined flow of the Current and Jacks Fork Rivers comes from springs, which account for their year-round, sustained flows.

Big Spring, one of the largest springs in the United States, has an average flow of approximately 287 million gallons of water per day. The impressive hydrogeologic character of OZAR karst landscape supports an amazing variety of natural features, including a spring system unparalleled in North America. The cave system is equally impressive with one of the highest densities of caves in any national park.

## 2.6 Local Climate

According to climate-data.org (<https://en.climate-data.org/north-america/united-states-of-america/missouri/ozark-16436/>), the Ozark lies on 363m above sea level. The climate in Ozark is warm and temperate. Ozark has a significant amount of rainfall during the year. This is true even for the driest month. The average annual temperature is 13.0 °C | 55.4 °F in Ozark. Precipitation here is about 42.9 inch per year. The driest month is January, with 1.9 inch of rain. With an average of 4.7 inch, the most precipitation falls in May. July is the warmest month of the year. The temperature in July averages 25.2 °C. January has the lowest average temperature of the year. It is -0.5 °C.

## 2.7 Sensitive Environments

Although USEPA considers all NPS lands to be sensitive environments, the definition under CERCLA is “a terrestrial or aquatic resource, fragile natural setting, or other area with unique or highly-valued environmental or cultural features.”

The U.S. Fish and Wildlife Service (FWS) Information for Planning and Consultation (IPaC) database (<https://ecos.fws.gov/ipac/>) was consulted to identify sensitive environments and protected species within the vicinity of the SAFRs. A seven square mile area surrounding the each SAFR was evaluated as potential habitat for Threatened and Endangered Species and the wildlife in the table below were identified

TYPE	NAME	STATUS	CRITICAL HABITAT
Mammals	Gray Bat, <i>Myotis grisescens</i>	Endangered	No
Mammals	Indiana Bat, <i>Myotis sodalis</i>	Endangered	Yes
Mammals	Northern Long-eared Bat., <i>Myotis septentrionalis</i>	Threatened	No
Birds	Red-cockaded Woodpecker, <i>Picoides borealis</i>	Endangered	No
Amphibians	Ozark Hellbender, <i>Cryptobranchus alleganiensis bishopi</i>	Endangered	No
Flowering Plants	Virginia Sneezeweed, <i>Helenium virginicum</i>	Threatened	No

<sup>1</sup> The area around Upper Current SAFR is a Critical Habitat for the Indiana Bat, *Myotis sodalis*.



The birds listed below are birds of particular concern either because they occur on the USFWS Birds of Conservation Concern (BCC) list or warrant special attention in the study areas.

BIRD NAME	BREEDING SEASON	UPPER CURRENT SAFR	BIG SPRING SAFR
Bald Eagle <i>Haliaeetus leucocephalus</i>	Breeds Sep 1 to Jul 31	No	Yes
Blue-winged Warbler <i>Vermivora pinus</i>	Breeds May 1 to Jun 30	No	Yes
Cerulean Warbler <i>Dendroica cerulea</i>	Breeds Apr 23 to Jul 20	Yes	Yes
Eastern Whip-poor-will <i>Antrostomus vociferus</i>	Breeds May 1 to Aug 20	Yes	Yes
Kentucky Warbler <i>Oporornis formosus</i>	Breeds Apr 20 to Aug 20	Yes	Yes
Prairie Warbler <i>Dendroica discolor</i>	Breeds May 1 to Jul 31	Yes	Yes
Red-headed Woodpecker <i>Melanerpes erythrocephalus</i>	Breeds May 10 to Sep 10	Yes	Yes
Rusty Blackbird <i>Euphagus carolinus</i>	Breeds May 10 to Sep 10	No	Yes
Wood Thrush <i>Hylocichla mustelina</i>	Breeds May 10 to Aug 31	Yes	Yes

Wetlands in each study area were evaluated using the National Wetlands Inventory (NWI) website. Wetlands associated with the Upper Current SAFR are shown on Figure 2-1. NWI identifies the adjacent Current River as 314 acres of riverine habitat classified as R3UBH. Wetlands associated with the Big Spring SAFR are shown on Figure 2-2. NWI identifies a 22.31-acre Freshwater Forested/Shrub Wetland habitat directly south of the SAFR classified as a PFO1A associated with Chilton Creek. Approximately 2.2 miles downgradient, Chilton Creek is the confluence of Chilton's Creek and the Current River. In this location, 14,243 acres of the Current River are classified as R2UBH. The following index defines the wetland classification systems for these wetlands.

- R: Riverine System includes all wetlands and deep-water habitats contained within a channel, with two exceptions: (1) wetlands dominated by trees, shrubs, persistent emergent, emergent mosses, or lichens, and (2) habitats with water containing ocean-derived salts of 0.5 ppt or greater.



- Subsystem Upper Perennial (3) is characterized by a high gradient. There is no tidal influence, and some water flows all year, except during years of extreme drought.
- Subsystem Lower Perennial (2) is characterized by a low gradient. There is no tidal influence, and some water flows all year, except during years of extreme drought. The substrate consists mainly of sand and mud. Oxygen deficits may sometimes occur.
- Class Unconsolidated Bottom (UB) includes all wetlands and deep-water habitats with at least 25% cover of particles smaller than stones (less than 6-7 cm), and a vegetative cover less than 30%.
- Water Regime Permanently Flooded (H) indicates water covers the substrate throughout the year in all years.
- Wetland classifications system identifies PFO1A wetlands as:
- System Palustrine (P) includes all nontidal wetlands dominated by trees, shrubs, persistent emergents, emergent mosses or lichens, and all such wetlands that occur in tidal areas where salinity due to ocean-derived salts is below 0.5 ppt. It also includes wetlands lacking such vegetation, but with all of the following four characteristics: (1) area less than 8 ha (20 acres); (2) active wave-formed or bedrock shoreline features lacking; (3) water depth in the deepest part of basin less than 2.5 m (8.2 ft) at low water; and (4) salinity due to ocean-derived salts less than 0.5 ppt.
- Class Forested (FO) characterized by woody vegetation that is 6 m tall or taller.
- Subclass Broad-Leaved Deciduous (1) are woody angiosperms (trees or shrubs) with relatively wide, flat leaves that are shed during the cold or dry season; e.g., black ash (*Fraxinus nigra*).
- Water Regime Temporary Flooded (A) indicates surface water is present for brief periods (from a few days to a few weeks) during the growing season, but the water table usually lies well below the ground surface for most of the season.

## 2.8 Summary of Previous Investigations

The NPS conducted investigations at three SAFRs in OZAR in 2006 (Terracon 2006) which included the Upper Current SAFR and the Big Spring SAFR, culminating in *Site Assessment Report, Small Arms Firing Ranges, Ozark National Scenic Riverways, Southern Missouri*, Terracon 2006 produced by Terracon Consultants, Inc., Omaha Nebraska. Terracon's investigation at each SAFR consisted of an initial visual assessment to visually delineate the extent of bullet fragments, and to identify areas of high bullet density. High density lead soil areas were then collected as discrete samples which were analyzed at a fixed laboratory for lead concentrations in soil.

### 2.8.1 Upper Current SAFR

Twenty-three soil samples were collected from across the Upper Current Firing Range site for visual and physical assessment and for laboratory analysis of total lead. Sampling locations are depicted in Figure 4 of Appendix A. Samples were also collected for background data. Soil samples were collected from the 0 to 6-inch depth at locations B-1 through B-23. Soil samples were collected from the 6 to 12-inch depth at locations B 12 through B-15 where projectiles (bullets and lead shot) were observed beyond a depth of 6



inches below ground surface. Sampling equipment was cleaned using an Alconox® detergent wash and de-ionized rinse water prior to beginning the project and between each sampling event.

### **Backstop**

The surface soil investigation revealed the highest levels of lead at the Upper Current Backstop. Five of the eleven soil samples collected contained lead bullets or fragments and also contained the highest total lead concentrations, which were in the range of 70.8 to 23,600 mg/kg.

### **Fallout Area**

The eight soil samples collected from the Fallout Area located beyond the Backstop did not contain bullets or fragments. Three of the eight samples analyzed for total lead ranged in concentration from 23.3 to 119 mg/kg.

### **Range Floor (between the firing line and the earthen backstop)**

One of the four samples collected from the Upper Current Range Floor contained one bullet. Three of the four samples collected were analyzed for total lead. Concentrations of total lead in the samples ranged from 31.3 to 279 mg/kg.

### **Background Area**

Two samples collected behind the firing line as background samples had total lead concentrations of 279 and 31.3 mg/kg total lead.

## **2.8.2 Big Spring SAFR**

The results of the visual assessment at the Big Springs Range revealed that spent lead bullets, bullet fragments, or lead shot were observed in eight of the 23 samples collected. Seventeen samples were analyzed for total lead. Sampling locations are depicted in Figure 10 of Appendix A. The concentration of lead in samples ranged from 11 to 7,360 mg/kg total lead. The highest concentration of lead in a sample where no bullets or fragments were observed was 786 mg/kg total lead.

### **Backstop/Fallout Area**

The surface soil investigation revealed the highest levels of lead at the Big Springs Backstop/Fallout Area. Four of the eight soil samples collected contained lead bullets or fragments and also contained the highest total lead concentrations, which were in the range of 73.4 to 7,360 mg/kg.

### **Range Floor**

Four of the twelve samples collected from the Big Spring Range Floor contained projectiles. Seven of the twelve samples collected were analyzed for total lead. Concentrations of total lead in the samples ranged from 24.2 to 2,120 mg/kg.



## **Background Area**

Two of the three background samples collected behind the target line contained total lead concentrations of 14.8 and 11.0 mg/kg.

### **2.8.3 Data Quality/Usability**

Data from the Terracon Site Assessment was collected in 2006 when both Sites were active SAFRs. Use of the SAFRs between 2006 and present as firing ranges has likely changed the conditions, making the previous data unreliable. In addition, the data quality objectives (DQOs) for the original study were based on continued use, whereas the current study is based on CERCLA site closure, requiring evaluation of long-term risk to human health and the environment. Based on this understanding, the previous data should be used qualitatively, to guide the current study but should not be used to quantitatively supplement the current study, which will be comprehensive, with the purpose of human health and ecological risk evaluation for lead in soil.

### **2.8.4 Preliminary Identification of Data Gaps**

Because both Sites have been used as active firing ranges between 2006 (Terracon 2006), the current study will assume conditions have changed at both Sites and all previously areas should be re-evaluated for risk assessment purposes.

Preliminary identification of data gaps include:

- A statistically relevant determination of lead concentrations in naturally occurring background soil;
- A statistically relevant determination of lead concentrations within the areas of concern (AOCs) at each Site (firing lines, target area berms, fallout areas;
- Characterization of the vertical extent of lead impacts to soil;
- Risk screening to characterize potential risks to human and ecological receptors at the Site; and
- Synthetic Precipitation Leaching Procedure (SPLP) analysis of on-site soils to determine whether leaching of lead from soil to groundwater is a transport pathway of concern.

### **2.8.5 Contaminants of Potential Concern and Contaminants of Potential Ecological Concern**

The only Contaminant of Potential Concern (COPC) and Contaminant of Potential Ecological Concern (COPEC) to be evaluated under the EE/CA is lead.

### **2.8.6 Media of Potential Concern**

The Media of Potential Concern (MOPC) at the Site are surface soil and subsurface soil to a depth of two feet.

Soil located on the Site has the potential to expose recreational visitors and site workers via direct contact exposure, as well as ecological receptors, including, birds, mammals, reptiles, plants and invertebrates.





## 2.9 Current and Future Property Use Scenarios

The Sites were previously used as active firing ranges. That use has been discontinued, and current Site activities will be focused on the foundational purpose of the Park. Ozark National Scenic Riverways (National Riverways) was established by an Act of Congress in 1964 (Public Law 88-492) to conserve and interpret unique scenic and other natural values and objects of historic interest, including preservation of portions of the Current and Jacks Fork Rivers in the Ozark Highlands of southeastern Missouri, in Shannon, Carter, Dent, and Texas counties. Within its boundaries are approximately 80,785 acres of river, forest, open field, and glade environments. Of that, 51,654 acres are federally owned, and 29,131 acres are in nonfederal ownership.

The National Riverways includes portions of the Current and Jacks Fork Rivers, providing 134 miles of clear, free-flowing, spring-fed waterways. Much of the area is underlain by dolomite that has given rise to numerous sinkholes, caves, and springs representative of classical karst topography. Up to 90% of the combined flow of the Current and Jacks Fork Rivers comes from springs, which account for their year-round, sustained flows. Big Spring, one of the largest springs in the United States, has an average flow of approximately 287 million gallons of water per day. The impressive hydrogeologic character of the National Riverways' karst landscape supports an amazing variety of natural features, including a spring system unparalleled in North America. The cave system is equally impressive with one of the highest densities of caves in any national park. The National Riverways lies within the Ozark Highlands, an important center of biodiversity in North America. The Ozark Highlands are home to a rich array of wildlife and plants, including endemic species that exist nowhere else in the world.

The Current and Jacks Fork Rivers have been designated as Outstanding National Resource Waters in Missouri.

## 2.10 Graphical Conceptual Site Exposure Model

Figure 2-3 presents the preliminary CSEM that has been used to develop the DQOs and identify data gaps associated with the current EE/CA.

The CSEM indicates that surface and subsurface soil are the exposure media for lead; therefore, the EE/CA Site Characterization will be focused on determining the nature and extent of any potential contamination in Site soil.

Potential receptors to soil exposure include:

- Human site visitors, adult and children;
- Human onsite workers, adults;
- Mammals and birds; and
- Plants and invertebrates.

Potential migration pathways for COPCs and COPECs in soil include:

- Soil to air via wind-blown particles; and
- Soil to terrestrial diet for ecological receptors.

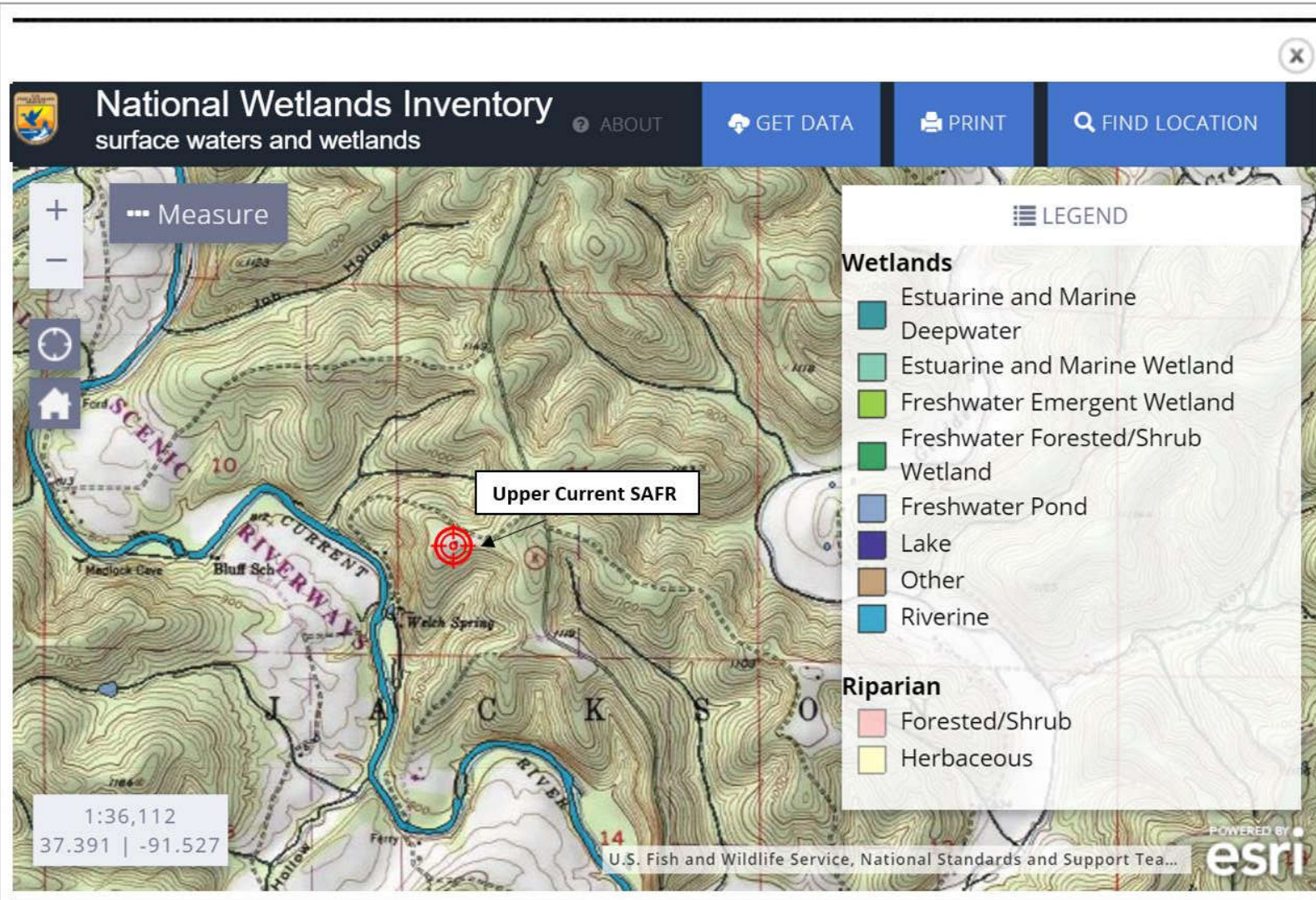


Exposure pathways for COPCs to human receptors include:

- Inhalation (ambient air/dust);
- Ingestion (surface soil); and
- Direct/dermal contact (surface soil).

Significant exposure pathways for COPECs to ecological receptors include:

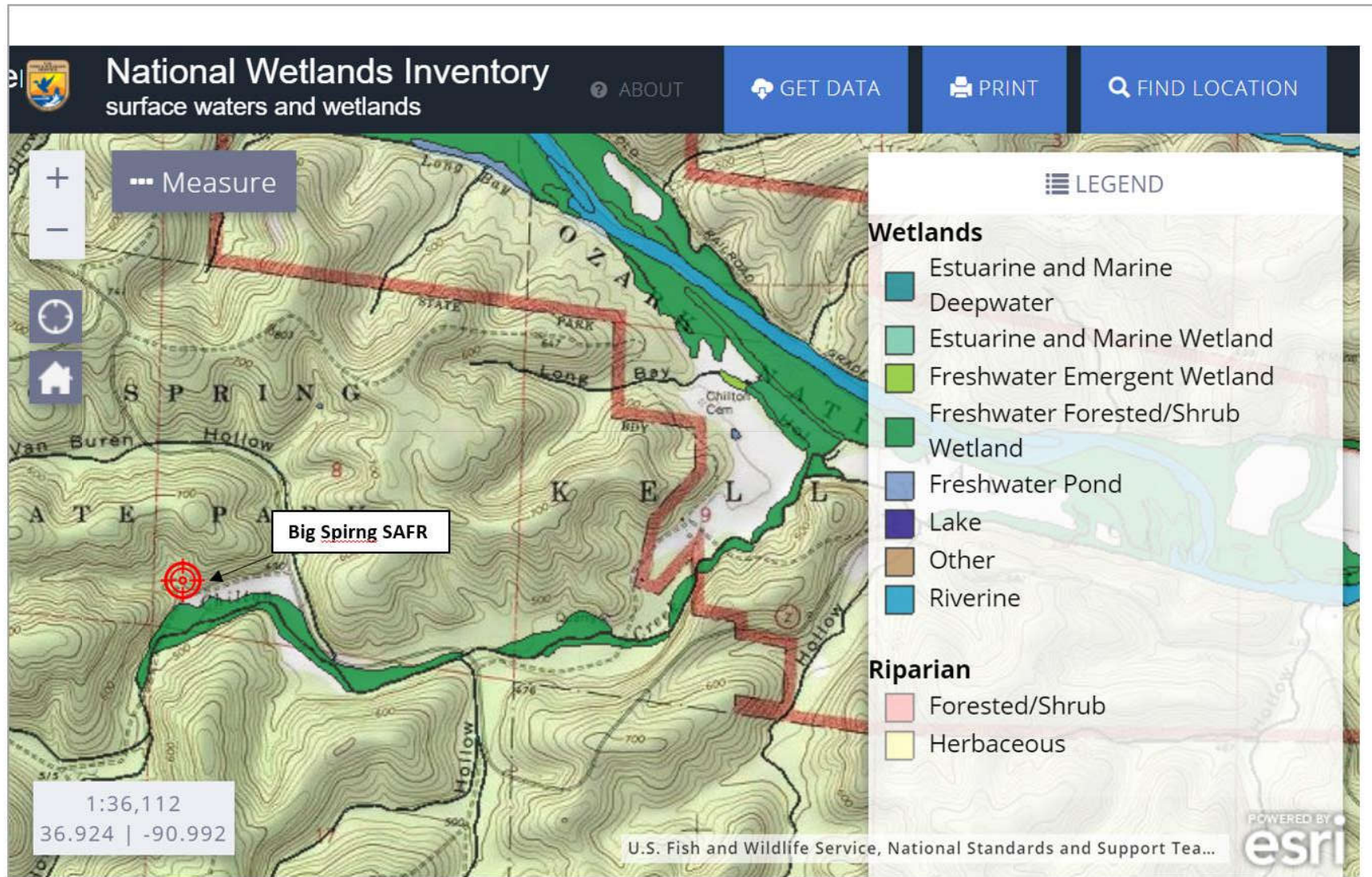
- Uptake/ingestion (surface soil);
- Direct/dermal contact (surface soil);
- Ingestion (terrestrial diet);
- Uptake/ingestion (subsurface soil) – plants and invertebrates only; and
- Direct/dermal contact (subsurface soil) – plants and invertebrates only.



**Figure 2-1** Upper Current SAFR, National Wetlands Inventory Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs

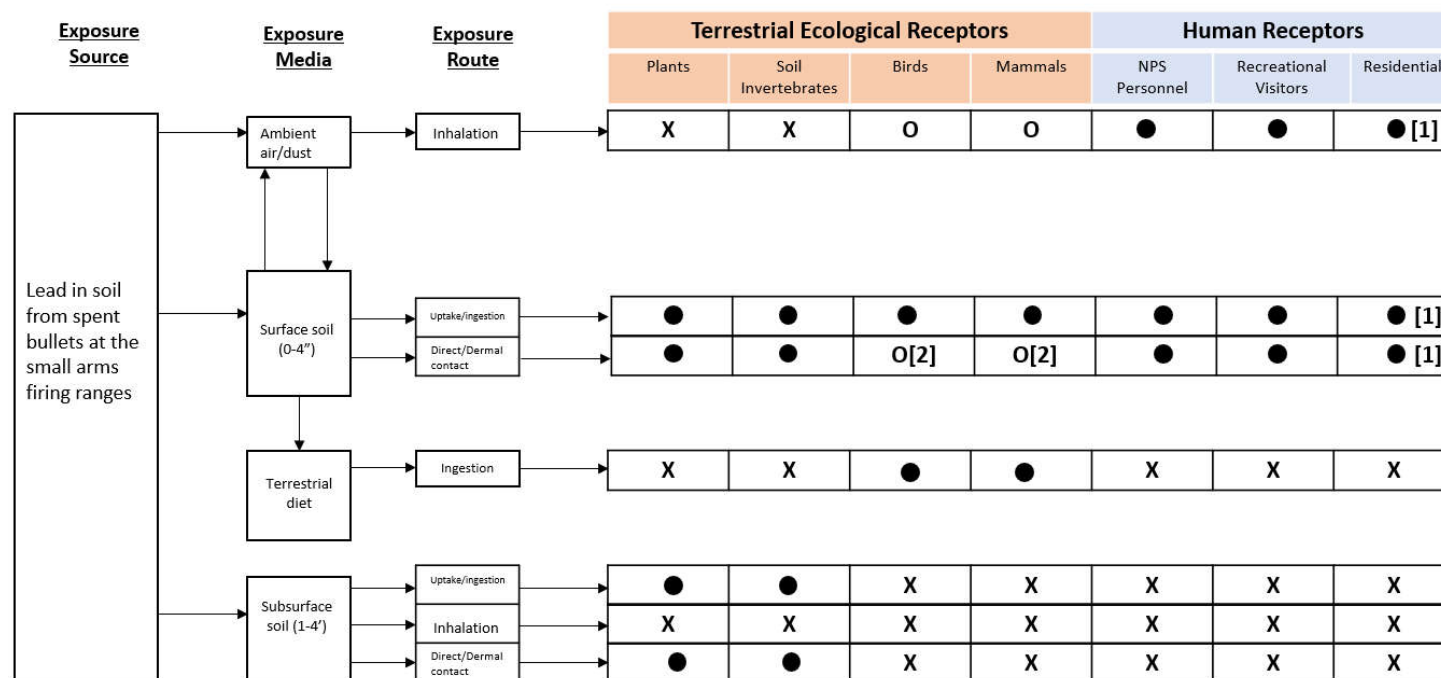






**Figure 2-2** Big Spring SAFR, National Wetlands Inventory Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs





#### LEGEND

- Pathway is believed to be complete and may be an important contribution to the total risk to the receptor
- O Pathway is believed to be complete but is unlikely to be a major contributor to the total risk to the receptor (in comparison to one or more other pathways that are evaluated)
- X Pathway is incomplete or believed to be negligible

[1] This scenario is considered highly unlikely; however, it will be evaluated for comparison purposes

[2] Burrowing animals may be exposed to surface and subsurface soils while digging both via incidental ingestion, inhalation, and dermal contact. However, available exposure and toxicity data are too limited to perform a quantitative evaluation of inhalation and dermal exposures and these pathways are considered negligible compared to ingestion.

**Figure 2-3 Conceptual Site Exposure Model**  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs







### 3. DQO PLANNING TEAM AND STAKEHOLDERS

The DQO Planning Team includes the primary decision makers and project team members, including risk assessors and remediation engineers, who will use the generated data.

Table 3-1 Planning Team Information				
Name	Role	Address	Telephone number	E-mail address
James Bissaillon	NPS Contracting Officers Representative (COR)	NPS, Interior Regions 3,4 & 5	440-665-0915	james_bissaillon@nps.gov
Eric Daniels	Chief of Resource Management	OZAR PO Box 490 Van Buren, MO	701-623-4466	eric-daniels@nps.gov
Jeff Hart	AI Project Manager	2801 Youngfield St., #240 Golden, CO 80401	Cell: 720 580 3453	jeff.hart@ap-in.com
John DeAngelis	AI Technical Support	2801 Youngfield St., #240 Golden, CO 80401	Cell: 303 246 8864	john.deangelis@ap-in.com
Michael Wicker	AI Geologist, field support and data review	2801 Youngfield St., #240 Golden, CO 80401	Cell: 435 363 6690	michael.wicker@ap-in.com

#### 3.1 Decision Makers

The decision makers have the ultimate authority for making final decisions based on the recommendations of the DQO Planning Team. The decision makers for this project are James Bissaillon (NPS, COR), Veronica Dickerson (NPS, Technical Lead), Eric Daniels (NPS, OZAR), and Jeff Hart (AI).

#### 3.2 Stakeholders

Stakeholders are parties who may be affected by the results of the investigation and/or persons who may later use the data resulting from the DQO process. Stakeholders for NPS lands may include tribal governments, states, non-governmental organizations, and other federal agencies. Stakeholder involvement must be considered within the context of the project (e.g., is this an enforcement case involving potentially responsible parties and confidential information).

### 4. DATA QUALITY OBJECTIVES



The DQO process specifies anticipated project decisions, the data types and quality required to support those decisions, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. USEPA Guidance on Systematic Planning Using the Data Quality Objectives Process (USEPA, 2006) were used to develop the DQOs.

The DQO process consists of the following seven steps, described in detail below:

1. State the Problem;
2. Identify the Goal(s) of the Investigation;
3. Identify the Information Inputs;
4. Define the Boundaries of the Investigation;
5. Develop the Analytic Approach;
6. Specify Performance or Acceptance Criteria; and
7. Develop the Plan for Obtaining Data.

The following sections detail each step in the DQO process for this investigation.

#### 4.1 State the Problem

Prior investigations at the Site (see Section 2.2) identified elevated lead concentrations in surface soil surrounding three on-site buildings, but data gaps were identified (see Section 2.2.4). Additional investigation is required to characterize the extent of lead contamination, to evaluate risk to Park Workers and Park Visitors, and ecological receptors, and to evaluate the leachability of lead in soil to groundwater.

The problem to be addressed by the current EE/CA is to characterize lead concentrations in Site soils, evaluate whether these concentrations pose risks to human and/or ecological receptors, and to evaluate the potential of lead in soil to leach to groundwater.

#### 4.2 Identify the Goal of the Investigation

The primary goal of the investigation is to collect sufficient environmental data to evaluate the extent of lead contamination to surface soil and subsurface soil from SAFR spent ammunition and to determine whether it poses potential risks to human and ecological receptors. Additionally, the potential for lead to leach from soil to groundwater will be evaluated.

##### 4.2.1 Principal Investigation Questions

Data collected from proposed sampling activities will support a decision-making process to determine risks to human and ecological receptors and evaluate remedial alternatives. The principal investigation questions are as follows:

**Principal Investigation Question 1:** What is the horizontal and vertical extent of lead in surface and subsurface soil at the Site?

**Principal Investigation Question 2:** What is the leaching potential of lead from soil to groundwater?



**Principal Investigation Question 3:** Do the concentrations of lead in Site soils present risks to human and ecological receptors?

### 4.3 Identify Information Inputs

The purpose of this step is to identify the data required to answer to the principal investigation question(s) listed in Section 4.2.1 and to determine which inputs require environmental measurements.

- Evaluate the quality and usability of existing sources of information and data that could be used to answer the principal investigation question(s).
- Identify the types of new information and data (e.g., information on specific analytes/contaminants) needed to answer the principal investigation question(s).

#### 4.3.1 Previous Data Usability

Previous investigations of lead impacts to the Sites were collected in 2006 (Terracon 2006) while the Sites were both active firing ranges. It is likely that additional firing range use since 2006 has changed the conditions at the Sites. Data was not collected for risk assessment; therefore, the Quality Control/Quality Assurance (QA/QC) sampling program was not robust as needed for risk assessment. In addition, discrete sampling was used, which is less supportive of cleaning up entire decision units. Prior data collected at the Site has been used to guide the sampling strategy of the current investigation but will not be directly used to evaluate the principal investigation questions.

#### 4.3.2 Data to be Collected During the Current Investigation

The new data required to answer the principal investigation questions are as follows:

- Background soil samples collected from an area at each SAFR not impacted spent bullets and fragments for analysis via EPA Method 6010 for lead using ISM methodology.;
- Surface soil samples (0 to 0.5 ft bgs) collected using ISM sampling methodology (ITRC 2012) within Decision Units (DU) developed to quantify risk and develop cleanup alternatives;
- Shallow subsurface soil samples (0.5 to 2 ft bgs) collected using ISM sampling methodology (ITRC 2012) within DUs developed to quantify risk and develop cleanup alternatives;
- Surface soil samples (0 to 0.5 ft bgs) collected using discrete sampling methodology developed to identify hotspots and lead exceeding screening levels in areas surrounding the firing ranges;
- Subsurface soil samples (0.5 to 2 ft bgs) collected using discrete sampling methodology developed to identify lead hotspots at depth to develop cleanup alternatives;
- A minimum of three ISM replicates will be collected at each SAFR to evaluate the statistical relevance of the data set, and from the background DU at each SAFR

Tables 4-1 and 4-2 identify the sampling locations, analytes, analytical methods, and QA/QC samples that will be collected.

### 4.4 Define the Boundaries of the Investigation





The objective of this step is to identify SUs and to define the spatial and temporal elements of the investigation area. Figure 4-1 and 4-2 shows maps of the sampling area(s). Practical constraints that could interfere with sampling also are identified. Implementing this step helps ensure the data are representative of the population. Spatial and temporal boundaries permit the identification of decision units (DUs), the smallest user-defined area(s) for which a decision will be made.

#### **4.4.1 Spatial Boundaries**

The study boundaries generally include the area surrounding each SAFR that may have been impacted spent bullet fragment, as well as an unimpacted background surface soil DU, located sufficiently away from the target area at each SAFR where Site-related lead impacts are not expected to migrate.

#### **4.4.2 Temporal Boundaries**

Temporal boundaries will be determined by the NPS in future contracting actions. The EE/CA field activities are tentatively scheduled for spring of 2021 and the contractual period of the contract is March 16, 2022.

#### **4.4.3 Decision Units**

DUs are the smallest user-defined area(s) for which a decision will be made (e.g., to cleanup or not cleanup) based on sampling. A DU may consist of one or more SUs. SUs are user-defined areas from which samples are collected to determine a representative concentration for that area. More detailed information regarding decision units is presented in the Plan for Obtaining the Data (Section 4.7).

DUs identified for the Site are shown in Table 4-1, and Figure 4-1, and are summarized below. DUs will be defined according to which SAFR Site.

The proposed DUs at the Upper Current SAFR include:

- DU-1: The impact berm;
- DU-2: The range floor immediately in front of the impact berm; and
- DU-3: Background.

The proposed DUs at the Big Spring SAFR include:

- DU-1: The range floor immediately in front of the impact zone;
- DU-2: The northern edge of the impact zone;
- DU-3: The central portion of the impact zone;
- DU-4: The southern edge of the impact zone ; and
- DU-5: Background.

#### **4.4.4 Sampling Units**

There are no SUs associated with SAFR to be sampled for the EE/CAs.



## 4.5 Develop the Analytic Approach

The purpose of this section is to define the analytic approach that will be used to answer the principal investigation question(s) and what screening values, or standards will be used.

### 4.5.1 Decision or Estimation Parameters

Because ISM characterization of lead concentrations in soil surrounding SAFR Target area is desired in order to develop remedial alternatives at each SAFR, ISM lead concentrations in soil will be compared directly to applicable screening levels at each sample location assuming the ISM results are the equivalent to a mean concentration of each subsample increment. Discrete samples will be collected and analyzed to evaluate contamination at depth potential contaminant migration away from the main target areas these results will be used to inform the development of remedial alternatives for the EE/CAs

#### Screening Levels

Collected surface and subsurface soil samples will be evaluated in comparison to appropriate screening levels for evaluation of risk to human and ecological receptors. Project Screening Levels (PSLs) and laboratory method detection limits (MDLs) and practical quantitation limits (PQLs) are shown in Table 4-3.

For human receptors, the following screening levels will be used:

- EPA Regional Screening Level (RSL) for residential exposure to lead in soil is based on blood-lead modeling for the adult and child resident (USEPA, 2020); and
- EPA RSL for industrial exposure to lead in soil is based on blood-lead modeling for the adult worker (USEPA, 2020).

For ecological receptors, the following screening levels will be used:

- NPS Ecological Screening Values (ESVs) for soil for COPEC selection that are established in the NPS Protocol for the Selection and Use of Ecological Screening Values for Non-Radiological Analytes (NPS, 2018) for terrestrial plants and invertebrates and mammals and birds.

Collected samples from each DU in the impacted or potentially impacted areas will also be compared to background soil samples. Background soil samples are described in Section 4.6.1, background reference location rationale is addressed in Section 4.7, and background sample collection methodology is presented in Section 5.

## 4.6 Performance or Acceptance Criteria

The purpose of this step is to establish the criteria needed to maximize the ability of the investigation to obtain the data needed to answer the principal investigation question(s) accurately and with confidence.



#### **4.6.1 Quality Assurance/Quality Control**

This section details the quality assurance/quality control (QA/QC) measures that will be implemented during the investigation to minimize variability, mitigate the potential for false positive and/or false negative error, and increase accuracy and defensibility in the collected data. QA/QC procedures apply to both the laboratory and field operations. Data Quality Indicators (DQIs) for field and laboratory operations are shown in Table 4.4.

##### **Laboratory Quality Assurance/Quality Control**

Pace Analytical Laboratory (Pace) will analyze a subset of collected soil samples for lead by EPA Method 6010. Pace is accredited through the National Environmental Laboratory Accreditation Program (NELAP). Pace's QC criteria are summarized in Table 4.5.

##### **Laboratory Quality Control Evaluation**

The following laboratory QC samples will be evaluated in the analysis of lead:

- Initial calibration verification (ICV);
- Calibration Blanks, Initial & Continuing (ICB & CCB);
- Laboratory control standard (LCS);
- Method blank;
- Continuing calibration verification (CCV); and
- Matrix spike and spike duplicate (MS/MSD).

##### **Field Quality Control Samples**

Field QC samples are collected and analyzed to quantify the precision, accuracy, representativeness, completeness and comparability DQIs. The following field quality control samples will be collected during the EE/CA.

- ISM Replicates at a minimum of one DU per SAFR and at each Background DU;
- Duplicate discrete samples at a minimum of 10 percent or one per SAFR;
- One equipment rinsate blank will be collected during each day and a minimum of one per SAFR; and
- One triple volume will be collected for an ISM and discrete sample in each SAFR to be evaluated in the laboratory using Matrix Spike/Matrix Spike Duplicate (MS/MSD) spiking procedures.

Field QC samples and performance criteria are shown in relation to DQIs in Table 4-4. Field QC samples in relation to sampling locations are shown in Tables 4-1 and 4-2.

##### **Background Samples**

Background samples are collected when naturally occurring or ambient concentrations of a contaminant or contaminants may be present at the Site being investigated and/or to delineate the contribution of contaminants from the Site versus off-site sources. Figure 4-1 and Figure 4-2 show the background sampling areas for the Upper Current SAFR and Big Current SAFR, respectively, and details about the





background soil sampling area are shown in Table 4-2 and Table 4-3, respectively. Background samples will be collected using ISM procedures to be statistically compatible with ISM environmental media sampling methods. The rationale for choosing appropriate background reference sample locations is presented in Section 4.6 and background sample collection protocols is presented in Section 5.

### **Decontamination Procedures**

Equipment decontamination at the Site will be limited to the stainless-steel incremental sampler or spoons used to collect the soil. This equipment will be sprayed with an Alconox detergent solution and distilled water rinse between the collection of each discrete sample. Any other equipment that is visibly contaminated will also require decontamination. Disposable, one-time-use plastic equipment will be used to the extent practical to minimize decontamination and prevent cross-contamination. Decontamination of sensitive instrumentation which requires disassembly of the instrument shall follow the procedures described by the manufacturer in the instrument manual.

### **Instrument/Equipment Testing, Inspection, and Maintenance**

A hand-held GPS will be used for documentation of sampling locations (1.5 to 3meter precision). This equipment will be tested and inspected in accordance with the manufacturers' specifications prior to the sampling event.

The project laboratory will follow the testing, inspection and maintenance procedures required by EPA-approved methods and as stated in the laboratory's SOPs.

### **Instrument/Equipment Calibration and Frequency**

AI will not be using equipment that needs to be calibrated in the field for this field effort.

### **Inspection/Acceptance of Supplies and Consumables**

The following procedures will be followed for all project supplies and consumables:

- All sample collection devices and equipment will be appropriately cleaned prior to use in the sampling projects;
- All sample containers will be certified pre-cleaned for the analytes of interest. Field personnel will note the information on the certificate of analysis that accompanies sample containers to ensure that they meet the specifications and guidance for contaminant-free sample containers for the analytes of interest; and
- The laboratory will follow the procedures in its SOPs for inspection/acceptance of supplies and consumables.

### **Special Training and Certification**

All field personnel and contractors conducting field investigations are required to have Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training that satisfies OSHA requirements under CFR 1910.120, supplemented by annual 8-hour refresher courses.



AI field personnel performing the sampling activities have obtained a minimum one-year experience in environmental field sampling of soil, water, and or groundwater, and familiarized with EPA guidance for sampling protocols, as well as the SOPs attached to this SAP. These protocols include lithologic logging, sample collection, sample processing, compositing, labeling, sample handling and shipping, and decontamination.

#### **4.6.2 Decision Error Limits and Uncertainty Evaluation**

The purpose of this study is to evaluate the extent of on-site lead contamination in relation to background soil concentrations and to determine potential risk to human and ecological receptors. Therefore, the primary decision errors that could potentially be drawn from the study would be:

1. Concluding that the source and/or sources of the lead contamination originate spent bullets and artillery when in fact the contamination is not significantly different than background levels (false acceptance error);
2. Concluding that an area or areas contain soils with lead concentrations that are above lead screening levels when they actually are not (false acceptance error);
3. Concluding that the source or sources of lead contamination are not significantly different from background soils when in fact they are (i.e., a false rejection error); or
4. Concluding that an area or areas are below lead screening levels when it/they actually pose unacceptable risks to human or ecological receptors (i.e., a false rejection error).

To answer the principal investigation questions (Section 4.2.1) and reduce decision errors and uncertainty around the study conclusion(s), sufficient data must be collected from the media of concern to make scientifically defensible conclusions.

The areas that must be sufficiently sampled include:

- Background soil; and
- Features and areas of environmental concern (i.e., berms, target areas, etc).

#### **4.6.3 Data Validation and Usability**

The data will be provided by the laboratory in Level 3 data reports, which will describe any QC issues that the laboratory encountered and what corrective action was taken to resolve the problems. QC issues that will be evaluated will include:

- Sample holding times;
- Initial and continuing calibration verification;
- Interference check sample for inorganics;
- Determination of bias (MS/MSD percent recovery);
- Precision (e.g., replicate analysis);



- Detection limits; and
- Confirm identification data.

Professional judgment shall be used to validate the overall data package. The EE/CA report will provide a Data Assessment section to describe the quality of the data based on the results of field and laboratory QC samples. This section will inform the user about data quality and limitations.

### **Data Verification**

The following steps will be taken to complete the data verification and validation for this SI report:

- Review data reports for transcription and typographical errors (e.g., 0.5 v. .05; ppb v. ppm);
- Determine if sampling protocols were appropriate;
- Compare field replicate samples;
- Review laboratory QC (e.g., laboratory blanks, method standards, spike recovery, duplicates);
- Summarize detection limits for non-detectable results;
- Review detection limits for positive but non-quantifiable data;
- Review sampling program design for assessing media variability;
- Review background concentrations to help identify site-specific contamination; and
- Delete unusable data, attach qualifiers to usable data, and explain limitations of qualified data.

## **4.7 Plan for Obtaining the Data**

AI will implement an ISM sampling strategy to determine a representative lead concentration within various DUs. ISM surface soil and subsurface soil samples and equipment blank samples will be analyzed for lead by EPA method 8020A. Samples will be collected by a two-person crew over a period of four days.

ISM sampling will be conducted in areas where removal actions are anticipated and in background areas to develop background threshold values (BTVs).

ISM samples will contain a minimum of 40 discrete samples within each DU. Replicates will be collected approximately 1/3 of the distance to the nearest pin flag in a north, east, and west direction (three Replicate increments per pin flag). Each replicate increment will be placed in a separate labeled 2-gallon Ziploc bag.

Soil samples will be labeled and stored in a cool shipping container until shipping is conducted. Sample holding times are 180 days and soil samples do not require cooling or preservation. Equipment rinsate blanks will be preserved with nitric acid.

At the Upper Current SAFR:

- DU1: The impact berm, three ISM Replicates;
- DU2: The range floor immediately in front of the impact berm, ISM Normal sample;



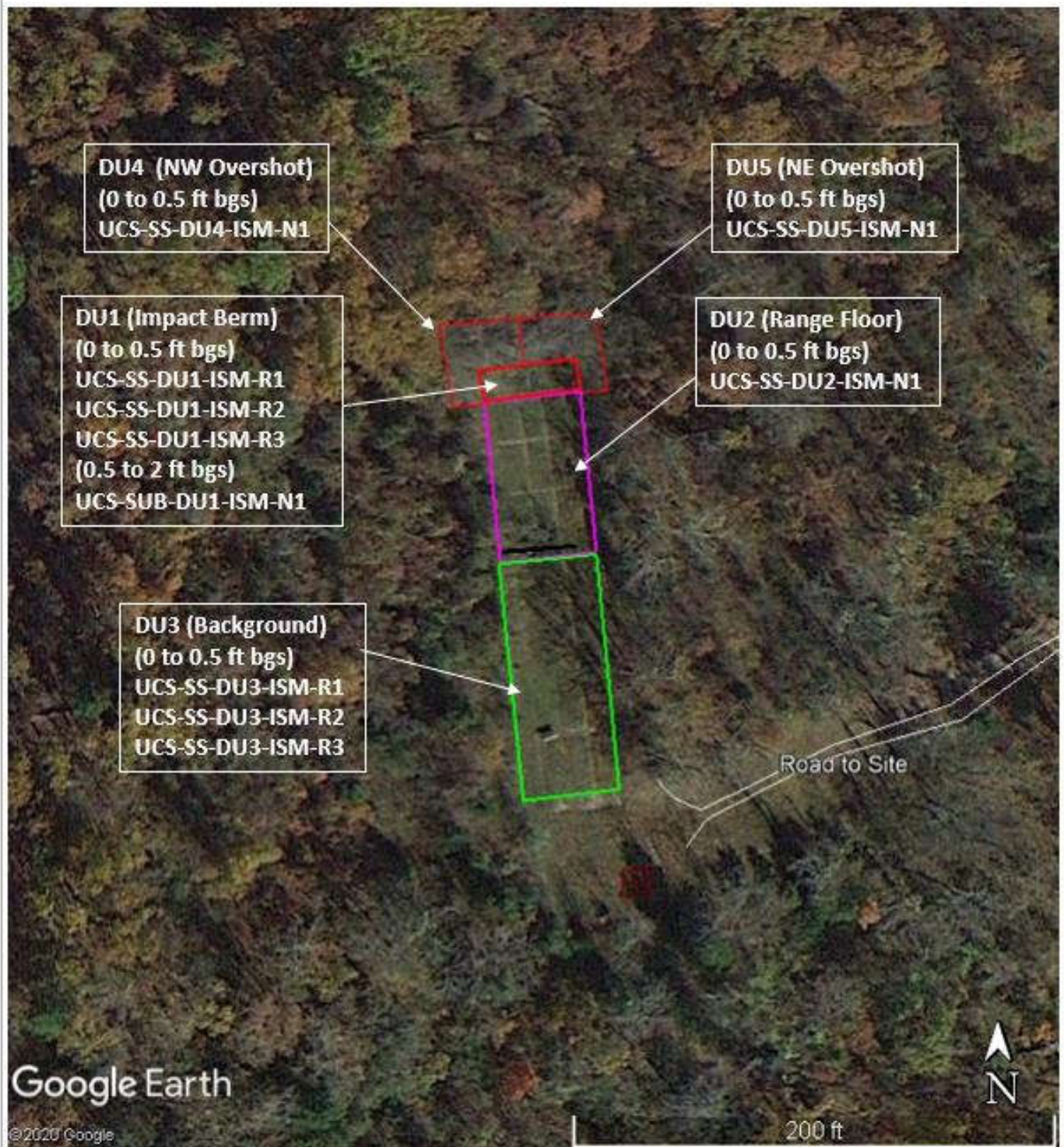


- DU3: Background, three ISM Replicates;
- DU4: Northwest overshot area adjacent to berm, ISM Normal sample;
- DU5: Northeast overshot area adjacent to berm, ISM Normal sample;
- One ISM sample will include triple volume for laboratory Matrix Spike/Spike Duplicate (MS/MSD) Quality Control (QC); and
- One equipment rinsate blank.

The proposed DUs at the Big Spring SAFR include:

- DU1: The range floor immediately in front of the impact zone, one ISM Normal sample;
- DU2: The northern edge of the impact zone, one ISM Normal sample;
- DU3: The central portion of the impact zone, three ISM Replicates;
- DU4: The southern edge of the impact zone, one ISM Normal sample;
- DU5: Background, three ISM Replicates;
- One ISM sample will include triple volume for laboratory MS/MSD QC; and
- One equipment rinsate blank.

Field QC samples and performance criteria are shown in relation to DQIs in Table 4-4. Field QC samples in relation to sampling locations are shown in Tables 4-1 and 4-2. Sample locations are shown Figure 4-1 and Figure 4-2.



**Figure 4-1** Upper Current SAFR Sample Location Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs







Figure 4-2 Big Spring SAFR Sample Location Map  
Sampling and Analysis Plan  
Current and Big Spring SAFR EE/CAs







Table 4-1 Project Screening Levels (Soils)										
Analyte	CAS Number	USEPA RSLs Residential Soil <sup>1</sup> (mg/kg)	USEPA RSLs Industrial SLs <sup>2</sup> (mg/kg)	NPS ESV (B&M) <sup>3</sup> (mg/kg)	NPS ESV (P&I) <sup>4</sup> (mg/kg)	Project Screening Level (mg/kg)	Project Screening Level Reference	Laboratory-specific		
								Method	PQL (mg/kg)	MDL (mg/kg)
Lead	7439-92-1	400	800	0.94	50	50	P&I ESV	6010B	0.5	0.208

<sup>1</sup> USEPA RSLs for residential exposure to lead, based on blood-lead modeling for child and adult residents (USEPA 2020)

<sup>2</sup> USEPA RSLs for industrial exposure to lead, based on blood-lead modeling for adult industrial workers (USEPA 2020)

<sup>3</sup> NPS COPEC selection ESVs for birds and mammals (NPS 2018)

<sup>4</sup> NPS COPEC selection ESVs for plants and invertebrates (NPS 2018)

CAS – Chemical Abstract Service

ESV – Ecological Screening Value

M – Mammals

MDL – Method Detection Limit

NPS – National Park Service

P&I – Plants & Invertebrates

PQL – Practical Quantitation Limit

PSL – Project Screening Level

RSL – Regional Screening Level

SL – Screening Level

USEPA – United States Environmental Protection Agency



Table 4-2 Sample Analysis Table – Upper Current SAFR (UCS)								
Sample Location		Media	Sample Method	QA/QC Type	Sample Identifier	Chemical/Physical Analysis		Lab QA/QC
						Lead EPA 6010B	Paste pH EPA 9054C	MS/MSD (5%)
Upper Current SAFR	DU1 (Impact Berm)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	R	UCS-SS-DU1-ISM-R1	X	X	
			ISM	R	UCS-SS-DU1-ISM-R2	X		
			ISM	R	UCS-SS-DU1-ISM-R3	X		
		Shallow sub-surface soil (SUB) (0.5 to 2 ft bgs)	ISM	N	UCS-SUB-DU1-ISM-N1	X		
	DU2 (Range Floor)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	N	UCS-SS-DU2-ISM-N1	X	X	X
	DU3 (Background)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	R	UCS-SS-DU3-ISM-R1	X		
			ISM	R	UCS-SS-DU3-ISM-R2	X		
			ISM	R	UCS-SS-DU3-ISM-R3	X		
	DU4 (NW berm Area)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	N	UCS-SS-DU4-ISM-N1	X		
	DU5 (NE berm Area)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	N	UCS-SS-DU5-ISM-N1			
	Field QC	Rinse Water	Pour over tool into container	EB	UCS -EB1-N1	X		

DU – Decision Unit

ISM – Incremental Sampling Methodology in accordance with ITRC 2012.

Lead by EPA 6010B – EPA Analytical Method using Inductively coupled plasma/mass spectroscopy (IC-MS)

pH -measurement of acid/base balance

QA/QC – Quality Assurance/Quality Control:

D – Duplicate discrete sample

EB – Equipment rinsate blank, Field quality control

N – Normal Sample

MS/MSD – Matrix spike/matrix spike duplicate

R- Replicate ISM Sample

SAFR – small arms firing range

SS – Surface soil

SUB – Subsurface soil

UCS – Upper Current SAFR Site



Table 4-3 Sample Analysis Table – Big Spring SAFR (BSS)								
Sample Location	Decision Unit	Media	Sample Method	QA/QC Type		Chemical/Physical Analysis		Lab QA/QC
						Lead EPA 6010B	pH	MS/MSD (5%)
Big Spring SAFR	DU1 (Range Floor)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	N	BSS-SS-DU 1-ISM-N1	X	X	X
	DU2 (North Impact Zone)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	R	BSS-SS-DU2-ISM-R1	X		
	DU3 (Central Impact Zone)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	R	BSS-SS-DU3-ISM-R1	X		
			ISM	R	BSS-SS-DU3-ISM-R2	X		
			ISM	R	BSS-SS-DU3-ISM-R3	X		
		Shallow sub-Surface soil (SUB) (0.5 to 2 ft bgs)	ISM	N	BSS-SUB-DU3-ISM-N1	X	X	
	DU4 (South Impact Zone)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	N	BSS-SS-DU4-ISM-N1	X		
	DU5 (Background)	Surface soil (SS) (0 to 0.5 ft bgs)	ISM	R	BSS-SS-DU5-ISM-R1	X		
			ISM	R	BSS-SS-DU5-ISM-R2	X		
			ISM	R	BSS-SS-DU5-ISM-R3	X		
	Field QC	Rinse Water	Direct Pour	EB	BSS -EB1-N1	X		

BSS – Big Spring SAFR Site

DU – Decision Unit

ISM – Incremental Sampling Methodology in accordance with ITRC 2012.

Lead by EPA 6010B – EPA Analytical Method using Inductively coupled plasma/mass spectroscopy (IC-MS)

pH -measurement of acid/base balance

QA/QC – Quality Assurance/Quality Control:

D – Duplicate discrete sample

EB – Equipment rinsate blank, Field quality control

N – Normal Sample

MS/MSD – Matrix spike/matrix spike duplicate

R- Replicate ISM Sample

SAFR – small arms firing range

SS – Surface soil

SUB – Subsurface soil





Table 4-4 Data Quality Indicators					
DQIs	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC for Field (F), Laboratory (L), or Both (F&L)
Precision	Soils	Lead (EPA 6010B)	RPD $\leq$ 30% for discrete field duplicates when $\geq$ the LOQ	Field duplicates (discrete samples)	F
Precision			RSD $\leq$ 3 for field ISM replicates when $\geq$ the PQL	Field replicates (ISM samples)	F
Precision			RPD $\leq$ 30% for both spiked field samples	MS/MSD	L
Accuracy			Spike recovery 80-120%	MS/MSD	L
Accuracy			Contamination – no target compounds $\geq$ the PQL	Field blanks, equipment blanks, trip blanks, instrument blanks	F&L
Accuracy			Contamination – no target compounds $\geq$ the PQL	Soil sampling rinsate	F
Representativeness			Appropriate sample design and SOPs developed through DQO process	Data verification and data validation, reconciliation with DQOs, and data usability evaluation will be conducted (Section 4.6.3 and Section 7.3)	F
Completeness			95%	Number of valid samples relative to field sample plan	F
Comparability			Appropriate sample design developed through DQO process; one laboratory and one analytical method used for each type of analysis; SOPs for sample collection and analysis will be generated and followed – data verification	Data verification and data validation will be conducted (Section 4.6.3) during the investigation and after data are received from the laboratory. Field audits will be conducted to ensure the FSP and SOPs are being followed.	F&L
Sensitivity			$\pm$ 20% at PQL	Laboratory standard blank at PQL	L

RPD – Relative percent difference

TAL metals – Total analyte list metals



Table 4-5 Laboratory Quality Control Criteria – EPA Method 6010B*				
QC Check	Explanation	Run Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	A quality control standard that is prepared in the same acid matrix as the calibration standards from a source independent of calibration standards.	Beginning of each run to verify calibration	90-110% (mid-level) 80-120% (low-level)	Terminate analysis, correct problem, recalibrate, reanalyze
Laboratory Control Standard (LCS)	An aliquot of reagent water or other matrix to which known quantities of the method analytes are added in the laboratory.	Per batch of 20 samples	±20% of true value	Reanalyze; if still not within acceptable limits, re-digest and reanalyze all associated samples after the last acceptable LCS.
Method blank	Reagent water taken through entire process of sample preparation and analysis	Per batch of 20 samples	Lower than the highest value for the following: RL 10% regulatory limit 10% lowest sample concentration in a batch	Re-run once; if still unacceptable, re-digest all associated samples and reanalyze.
Continuing Calibration Verification (CCV)	Mid-range calibration standard	Every 10 samples, and at the end of each analytical run	±20% of true value	Terminate analysis, correct problem, recalibrate, reanalyze
Calibration Blanks, Initial & Continuing (ICB & CCB)	Same concentrations of same acids used to prepare final dilutions of calibrating solutions	Prior to sample analysis, following every 10 samples and at the end of the analytical set	< PQL	Terminate analysis, correct problem, recalibrate, reanalyze
Matrix Spike & Spike Duplicate ("S" or "SD" following sample ID)	Two aliquots of field samples specific to this project (not field blank) fortified at 10–100 times the PQL for each target analyte	5% of field samples	80–120 % Recovery	If MSD same as MS recovery and LFB is acceptable, assume matrix effect and record in narrative. If LFB also unacceptable, re-prepare and re-analyze LFB and QC sample set
Post-digestion spike	Analyte spike added to portion of prepared sample or its dilution to	To be performed if MS/MSD recovery outside 80-120%	80–120 % Recovery	Run dilution test (below). If both MS/MSD and post-



	produce between 10x min level and 10x max level of instruction detection limit			digestion spike fail, matrix effects are confirmed.
Dilution Test	Fivefold dilution of original sample	To be performed if post-digestion spike recovery outside 80-120% or if analyte concentration is a factor of 100 above LOD after dilution per EPA Method	±10% of original determination	Report potential chemical or physical interference

\*Note: ICP-MS calibration must be performed daily, prior to sample analysis. Correlation coefficient must be  $\geq 0.998$  for all analytes of interest. All reported values must fall between lowest and highest calibration standards (diluted if necessary).





## 5. FIELD SAMPLING PLAN

The Field Sampling Plan (FSP) guides the collection of data. Within the FSP, the sampling design is essential for ensuring the samples collected are representative of the decision area and the media being sampled and characterized.

Environmental Sampling SOPs developed for the EE/CA are provided in Appendix C and listed as follows:

- ISM soil sampling (AI-WES-05);
- Sample Handling (AI-WES-11);
- General field measurement parameters (AI-WFM-01);
- Equipment decontamination (AI-WFO-16);
- General record keeping (AI-WRK-01);
- Logbook (AI-WRK-02); and
- Field data forms (AI-WRK-03).

### 5.1 Soil Sampling

This section describes the soil sampling activities that will be conducted under this investigation. At both Sites, both discrete and ISM soil sampling will be conducted in accordance with the ISM techniques based on guidance from the ITRC (ITRC 2012), as well as the DQOs and SOPs presented in this SAP. ISM is a structured sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling. ISM provides representative samples of specific soil volumes defined as DUs by collecting numerous increments of soil that are combined, processed, and subsampled according to specific protocols. The sampling density afforded by collecting many increments, together with the disciplined processing and subsampling of the combined increments, in most cases yields more consistent and reproducible results than those obtained by more traditional (i.e., discrete) sampling approaches.

#### 5.1.1 Soil Sampling Locations

Soil sampling locations are summarized in Tables 4-2 and 4-3, depicted in Figures 4-1 and 4-2, and further described as follows:

Lead will be analyzed by EPA Method 6010B in the contract fixed laboratory. Soil pH will be analyzed by Paste pH EPA Method 9045C.

#### Upper Current SAFR

Table 4-2 and Figure 4-1 summarize the following:

- Impact Berm (DU1), surface soil (0 to 0.5 ft bgs) and shallow subsurface soil (1 to 2 ft bgs):



- Three ISM Replicates will be collected from surface soil at DU1, analyzed for lead and one of these Replicates will also be analyzed for Paste pH ; and
  - One ISM Normal sample will be collected from shallow subsurface soil at DU1, analyzed for lead.
- Range Floor (DU2), surface soil (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU2, analyzed for lead and for Paste pH .
- Background (DU3), surface soil (0 to 0.5 ft bgs):
  - Three ISM Replicates will be collected from surface soil at DU1, analyzed for lead.
- NW Berm Overshot Area (DU4) surface soil samples (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU4, analyzed for lead.
- NE Berm Overshot Area (DU5) surface soil samples (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU5, analyzed for lead.
- One equipment rinsate blank will be collected from digging tools using deionized water poured over a decontaminated tool, directly into a 250-milliliter (ml) poly container.

### Big Spring SAFR

Table 4-3 and Figure 4-2 summarize the following:

- Range Floor (DU1), surface soil (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU2, analyzed for lead and for Paste pH .
- Northern Impact Berm (DU2), surface soil (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU2, analyzed for lead.
- Central Impact Berm (DU3), surface soil (0 to 0.5 ft bgs) and shallow subsurface soil, (1 to 2 ft bgs):
  - Three ISM Replicates will be collected from surface soil at DU3, analyzed for lead and one of these Replicates will also be analyzed for Paste pH ; and
  - One ISM Normal sample will be collected from shallow subsurface soil at DU3, analyzed for lead.
- Southern Impact Berm (DU4), surface soil (0 to 0.5 ft bgs):
  - One ISM Normal sample will be collected from surface soil at DU4, analyzed for lead and for Paste pH .
- Background (DU5), surface soil (0 to 0.5 ft bgs):
  - Three ISM Replicates will be collected from surface soil at DU5, analyzed for lead.



- One equipment rinsate blank will be collected from digging tools using deionized water poured over a decontaminated tool, directly into a 250-ml poly container.

### **5.1.2 Soil Sampling Protocol**

Detailed protocol for soil sampling is provided in the SOPs in Appendix B. The majority of the collected soil samples will be collected according to ISM protocols (ITRC 2012). It is important that these protocols be followed for collection of increments properly and consistently to minimize variability in laboratory results that could be potentially caused by inconsistent sampling and processing techniques.

The following procedure will be used to collect surface and shallow subsurface soil samples, which includes natural soil, tailings, waste rock, and mixtures of these materials:

- If present, carefully remove the top layer of vegetation or organic matter to the desired sampling depth interval with a pre-cleaned spade;
- Obtain the sample from less than 6-inch depth (both discrete background and composite increments) and place into a plastic-lined bucket;
- A minimum of 40 equally spaced, equal volume increments will be required for ISM samples;
- Particles greater than about 1 cm are excluded from the initial collection procedure. Gravel, rock, and vegetation will be removed as practical from composited samples and discrete background samples as part of the homogenization process;
- ISM soil samples will be placed into a 1-gallon Ziplock bag without homogenization or subsampling in the field, labeled according to the numbering scheme described in Section 5.2.2;
- Discrete soil samples will be placed into laboratory-provided sampling jars with the cap tightly secured and labeled according to the numbering scheme described in Section 5.2.2; and
- Disposable plastic liners will be discarded and replaced between separate samples to prevent cross contamination. Sampling equipment will be decontaminated per Section 4.5.4.

During field sampling activities, AI will record surface conditions that may affect chemical analyses on a Soil Field Sampling Report (Appendix D). These conditions may include:

- Approximate distance to roadways and access roads;
- Observations of seepage;
- Soil type (clay, silt, sand, gravel);
- Site features, such as nearby drainage outfalls;
- Visual observations that will discriminate impacted soil from unimpacted soil, such as color, gradation, and/or proximity to mine features, such as mills, retorts, adits, tailings piles, and waste rock piles;
- Evidence of dumping or spillage of contaminants; and
- Soil discoloration, unusual condition of growing plants, and stressed vegetation.





### **5.1.3 Soil Sampling Health and Safety**

A HASP will be prepared prior to the initiation of field activities that details all health and safety requirements for the soil sampling activities to be carried out in the course of this EE/CA. The HASP will address all soil sampling related tasks, the potential hazards associated with each task, and the control measures and or personal protective equipment (PPE) implemented to prevent or mitigate potential hazards.

### **5.1.4 Soil Field Measurements**

Field measurements for soil are not anticipated for this investigation.

### **5.1.5 Soil Analytical Measurements/Methods**

Table 5-1 describes analytical methods, container types, soil volume requirements, preservation and holding times to correspond with laboratory requirements.

## **5.2 Sample Handling and Chain-of-Custody**

Sample custody will be maintained and documented throughout collection, shipping, analysis, and disposal of the sample. Samples will not be left unattended unless properly secured. A chain-of-custody (COC) form will be used to summarize the samples collected and analyses requested. The COC tracks the samples between their release from the field to the initial receiving laboratory by having each COC signed by the person relinquishing control of the samples and the person taking possession of them. The original COC will accompany the samples; the person releasing custody will keep a copy. Upon receipt at the laboratory, the COC will be signed by the person receiving the samples. The following information will be recorded on each COC:

- Project name;
- Sample identification;
- Sample type (grab or composite);
- Date and time of collection;
- Preservation technique;
- Analyses to be performed;
- Identification of sample collector;
- Shipping destination; and
- Signature and dates of all sample custodians.



Immediately upon receipt by the laboratory, the condition of samples will be assessed and documented. The contents of the shipping container will be checked against the information on the custody form for anomalies. If any discrepancy is identified, or if laboratory acceptance criteria or project-specific criteria are not met, the laboratory must contact the Project Manager for resolution of the problem. The discrepancy, its resolution, and the identity of the person contacted will be documented in the laboratory's file and copies of the documentation will be included in the final report. The following conditions may cause sample data to be unusable and must be communicated to the laboratory team leader:

- The integrity of the samples is compromised (e.g., leaks, cracks, grossly contaminated container exteriors or shipping cooler interiors, obvious odors, etc.);
- The identity of the container cannot be verified;
- The proper preservation of the container cannot be established;
- Incomplete sample custody forms (e.g., the sample collector is not documented, or the custody forms are not signed and dated by the person who relinquished the samples); and
- Required sample temperatures were not maintained during transport.

The laboratory will also verify that sample conditions, amounts, and containers meet the requirements for the sample and matrix. A unique sample identifier will be assigned to all containers for each sample received at the laboratory, including multiple containers of the same sample.

### **5.2.1 Sample Designation**

The characters of the sample location identifier shall be divided as follows:

- The first four characters are the site identifier, followed by a “-“:
  - UCS – Upper Current SAFR; and
  - BSS – Big Spring SAFR.
- The next two to three characters will identify the media type, followed by a “-“:
  - SS = Surface Soil; and
  - SUB = Surface Soil and Shallow Subsurface Soil.
- The following three characters will identify the decision unit (DU), followed by a “-“:
  - DU1 = Decision Unit 1;
  - DU2 = Decision Unit 2; and
  - DU3 = Decision Unit 3.
- The following three characters will identify the sampling and method, followed by a “-“:
  - ISM = ISM sampling methodology and laboratory preparation required; and
  - DIS = discrete sampling methodology and laboratory preparation required.
- The last two characters will QA/QC type and serial number:
  - N1 = Normal Sample 1;
  - R1 = Replicate sample 1;



- D1 = Duplicate sample 1; and
- E1 = Equipment rinsate sample 1.

Examples:

- UCS-SS-DU1-ISM-R3 is the Upper Current SAFR (UCS) surface soil (SS) collected at the Impact Berm (DU1) by ISM and is the third Replicate (R3) of the set.

Samples collected with triple volume for MS/MSD analysis will be designated on the Chain of Custody and will be collected as described in Tables 4-2 and 4-3. The sample label will be unchanged, but the Chain of Custody will indicate triple-volume for all analyses and will include in the comments box “Triple volume for Lab MS/MSD”.

### **5.2.2 Sample Labeling**

Sample labels will include the following information:

- Sample ID as indicated above;
- Sample date and time; and

Sample labels will be filled out using fine-tipped ink marker.

### **5.2.3 Documentation and Records**

AI SOP AI-WRK-01 details protocols for general record keeping, including the Site field logbook and standardized field forms maintained at the Site. Records shall be kept for all activities associated with the field activities, as a means to maintain full documentation of project QA/QC procedures and compliance. In general, all documents shall be completed in permanent black ink. Errors shall be corrected by crossing them out with a single line and then dating and initialing. The use of correction fluids shall not be allowed. The documents used during the field investigation shall remain on site (if possible) during the entire effort so that they can be reviewed by interested parties. Forms shall be kept organized and in a central file also located on site, if applicable. Examples of standardized field forms are attached to the SOPs in Appendix D.

## **5.3 Investigative-Derived Waste Handling**

Investigative-derived waste (IDW) generated at the Site will generally consist of:

- Soil cuttings and residual soil;
- Disposable sampling equipment (plastic scoops, spatulas, core samplers, used DPT acetate sleeves);
- Disposable PPE (latex gloves, ear plugs);
- Miscellaneous (used paper towels, baggies, disposable wipes); and



- Wash and rinsate water.

IDW generated during the field investigation shall be minimized. Generation of a significant amount of IDW is not anticipated and will primarily consist of paper towels, wet wipes, disposable gloves, and non-adhesive backing from sample labels. These IDW will be placed in plastic bags on-site and transported off-Site for proper disposal.

Soil cuttings will be minimal as most samples are surface soil samples. Thirty shallow excavations will be sampled from 0 to 2 ft bgs for the DU3 (Central Impact Zone). Soil cuttings from these borings will be utilized and sent to the lab for subsampling..

Decontamination of equipment will be minimal because sampling equipment that will contact the soil samples will be single-use and will be disposed of after each ISM or discrete sample is collected. Digging tools will be sprayed inside a 5-gallon bucket with Alconox-water and distilled water and the water will be allowed to evaporate onsite.

## 5.4 Health and Safety

The HASP (AI 2020) details all health and safety requirements for all soil sampling related tasks, the potential hazards associated with each task, and the control measures and or PPE implemented to prevent or mitigate potential hazards.

The AI sampling team will meet and review the HASP before beginning field activities. In addition, daily tailgate safety meetings will be conducted before each work shift to document review of safety hazards and discuss changing conditions and/or work tasks.





Table 5-1 Sample Handling Table							
Sample Matrix	Analysis	Analytical Method	Container Type	No. Containers	Required Vol.	Preservation	Technical Holding Time
Surface & shallow subsurface soil	Lead	EPA Method 3050B (Digestion) EPA Method 6010B (ICP-MS)	Ziplock bag (ISM) 4 oz glass jar (discrete)	1	1 kg (ISM) 1 g (discrete)	None	180 days
Surface & shallow subsurface soil	pH	EPA 9045D	Ziplock bag (ISM) 8 oz glass jar (discrete)	1	1 kg (ISM) 20 g (discrete)	None	180 days
Equipment Rinsate Blank	Lead	EPA Method 6010B (ICP-MS)	250-ml Poly container	1	250-ml	Nitric acid	180 days



## 6. DATA MANAGEMENT

The success of an environmental investigation relies on data and the ability to make accurate data interpretations. For good interpretations to be made, it is critical that data are:

- Of known quality;
- Reliable;
- Aggregated in a manner consistent with their prime use; and
- Accessible to a variety of users.

The data management plan for this program is established to enable data to be traced from their generation to their final use or storage and includes mechanisms for detecting and correcting errors. Data management personnel are identified in Table 6-1 and described below.

- Field Staff will collect samples, take field measurements, and record data. They will assemble data files (including raw field data, routine QC checks, data flags, and sampler comments) and submit these files to the Field Coordinator for verification.
- The Field Coordinator will review the field data records for completeness and correct any transcription errors, verify conformance with field SOPs, submit analytical samples to the laboratory, and submit validated field data records to the PM.
- The Laboratory will confirm the accuracy of the analysis request sheets, measure and record receiving information, and perform the requested analyses in compliance with the DQOs specified in the SAP, the laboratory QAM, and method-specific SOPs. It will provide the QAO and PM with laboratory validated sample results, electronic data deliverables, a copy of the completed chain-of-custody forms, and an explanation for any QA/QC anomalies in the analytical data.
- The Quality Assurance Officer (QAO) will perform routine independent reviews of data to ensure the project's DQOs are met and submit a report with the findings and any recommended corrective actions to the PM.
- The Project Manager (PM) will review the Field Coordinator and QAO's reports to identify problems that need to be rectified, initiate adjustments to the field and analytical program as necessary, and review conformance with the data management requirements.

### 6.1 Data Recording

All data input will be checked for accuracy and completeness and will have undergone review and approval by the QAO prior to use on the project. The AI team will receive a Level 3 report package from the laboratory. A Laboratory Level 3 report consists of the following:

- Cover letter/sheet with authorized project manager signature;
- Sample receipt summary;



- Case narrative (if needed);
- Analytical results summary;
- Batch QC data summary (method blank, LCS, batch MS/MSD);
- Data qualifier definitions and legend; and
- Field chain-of-custody form and sample receipt checklist.

The QAO will validate the report internally, after which an electronic spreadsheet will be developed. The hard copy data may contain professional judgment changes implemented by the laboratory compared to the preliminary electronic spreadsheet data. The AI team will then compare the electronic version of the data to the validated hard copies and make any necessary changes. The PM will be responsible for maintaining the integrity of the data and fulfilling requests for data access.

All measurements taken during this project will be identified by source and type to avoid ambiguity and ensure traceability. Field data will be entered into a computerized data management system and maintained under the supervision of the PM.

## **6.2 Data Transmittal**

Microsoft Excel will be used for the transmittal of data. Data are tracked via the laboratory's information management system (LIMS).

## **6.3 Data Tracking**

While in the laboratory, this project will be tracked via the LIMS. Following submittal of the data to the AI team, Microsoft Excel will be used to track sample numbers and analytical results.

## **6.4 Data Storage and Retrieval**

The data will be stored in electronic form in the laboratory's LIMS, as hard copy printouts of the instrument data, and as electronic copies of the hard copy data. AI will store project data files electronically on a secure computer with backup storage on the cloud. Laboratory records will be retained by the laboratory for a minimum of five (5) years. Project records will be retained by the AI team for a minimum of five (5) years.



Table 6-1 Data Management Personnel				
Name	Role	Address	Telephone No.	Email
John DeAngelis	AI Field Coordinator	2801 Youngfield Street, Golden, CO 80401	Office: 720-580-3457 Cell: 303-246-8864	john.deangelis@ap-in.com
Chris Ward	Pace Laboratories	12065 Lebanon Rd Mount Juliet, TN 37122	Office: 615-773-9712	chris.ward@pacelabs.com
Chelsea Maxfield	AI Quality Assurance Officer	345 Bobwhite Ct., Ste 230 Boise, ID 83706	Office: 360-362-0405 Cell: 360-941-6298	chelsea.maxfield@ap-in.com
Jeff Hart	AI Project Manager	2801 Youngfield Street, Golden, CO 80401	Office: 720-580-3453 Cell: 720-884-7404	jeff.hart@ap-in.com





## 7. ASSESSMENT AND OVERSIGHT

This section describes the measures that will be employed to ensure that this SAP is implemented properly.

### 7.1 Data QC Review

The QAO will review the laboratory's report package to ensure the project data has been recorded, transmitted, and processed correctly. The QAO will discuss any problems or discrepancies with the laboratory for resolution and/or documentation. The data review will include the following activities:

- An examination of project data, identifying errors in data entry, storage, calculation, reduction, transformation, or transcription.
- An examination to ensure all required sample information is documented and available in preparation for the verification, validation, and assessment process. This includes pertinent project information concerning blanks, matrixes, temperature requirements, duplicates, preservatives, shipping dates, holding times, chain-of-custody records, etc.
- An examination to identify if all required non-direct measurement data (existing data) information and supporting documentation, as required by the project SAP, have been received and are available for the verification and validation process.
- A completeness check to determine if any data deficiencies exist, such as missing data or compromised data integrity, due to issues such as loss in acquisition, storage, or processing.
- An examination to ensure all necessary analytical laboratory support documentation, as set forth and stipulated in the project SAP, has been received from the applicable laboratories.
- An examination to identify programming and/or software related errors, if applicable to the project.

#### 7.1.1 Data Verification

Data will be verified by the QAO with the focus of identifying all requirements specified, associated procedures, and project contractual requirements have been met. Data verification will at the minimum, include the following activities:

- Verification that all data completeness criteria, as stated in this document, have been satisfied. This shall include items such as the number of samples, number of QC samples such as spikes and duplicates, and chain-of-custody record continuity.
- Verification that the required analytical methods, as listed, correspond to the analytical methods employed by the laboratory, as recorded in laboratory reports.
- Verification that requirements relative to laboratory analytical support documentation have been satisfied by the reporting laboratory, including the correct application of data qualifiers.



- Verification that all supporting information and documentation for non-direct measurement data (existing data) meet the requirements. If not, identify any limitations or restriction on the use of such data.
- Verification that data and sample collection practices adhered to procedural requirements, to include a review of project logs and field notes, as applicable.
- Verification that sample handling activities conform to requirements. Examples include sample shipment timelines, sample holding times, preservatives, number of samples obtained, duplicate or split sample frequency, and chain-of-custody documentation.
- Verification that any remaining or unique project or procedural requirements have been met, and if not, determine the extent to which these requirements failed to be achieved. Determine and document any limitations on the use of the project data.

### **7.1.2 Data Quality Assessment**

Data quality assessment (DQA) will be performed in accordance with this document. Additional guidance for conducting data assessment can be found in EPA QA/G-9R or EPA QA/G-9S (EPA, 2006a; EPA, 2006b).

The DQA will be performed (at a minimum) by the project QAO to determine if the project data set is of the right type, quality, and quantity to achieve the objectives of the project and can confidently be used to make an informed decision. Information and findings associated with the project data review, verification, and validation efforts shall be considered during the data assessment process.

When DQOs are not met, the PM will discuss appropriate corrective actions with project staff, project management, and with the analytical laboratory. Corrective actions may be initiated to suggest improvements to data collection activities, data and sample handling techniques, internal laboratory quality procedures, etc., to solve quality issues.

If the project QAO decides the project data do not meet the project needs or DQOs and/or if the conclusions drawn from the data do not appear to be reasonable, the project QAO shall immediately report such findings to the NPS.

### **Peer Review**

An internal peer review will be completed by the AI team on all data sets, memoranda, and reports.

### **7.1.3 Field Audit and Response Actions**

Field audits may be conducted to evaluate field investigations sampling procedures to ensure that field protocols are being followed and standard forms are being utilized and maintained. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file. At the beginning of each week and at the completion of the site work, project file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been



taken and that corrective action reports are attached. If corrective actions have not been taken, the project manager will be notified to ensure action is taken.

The Project Field Team Leader will be responsible for reviewing all field forms and ensuring all SOPs are followed. Field forms completed field forms and chains of custody forms will be emailed to the Project Manager at the end of each day, including a daily field summary report. The Project Field Team Leader and Project Manager will review the daily completed work along with the planned to complete work and identify the work to be completed for the following day. Work will continue in this manner until all work is completed and signed off by the Project Manager and Field Team Leader.

#### **7.1.4 Laboratory Audit and Response Actions**

All laboratory data packages will be verified for completeness and technical accuracy by the laboratory performing the work. Data packages will then be reviewed by the AI team for completeness. Corrective Action reports will be reviewed to ensure all corrective actions were completed successfully. These will be documented in writing by the Laboratory PM.

### **7.2 Quality Assessment Reporting**

This section defines what QA reports will be required. Include the frequency with which they will be generated, who is responsible for generating them, and who receives the reports.

#### **7.2.1 Field Data Verification**

The QAO will provide a field QA/QC report to the NPS COR within one week of the end of the field effort to document the activities in the field, which will include a daily field activities report, and a complete list of chain of custody forms for the project which will document that all samples were collected and received by the laboratory and confirm that they were received in good condition for analysis by the laboratory.

#### **7.2.2 Laboratory Data Corrective Action Reports**

The Laboratory PM shall contact the AI PM within 24 hours of determination of a laboratory noncompliance issue that will require a corrective action via email, and if necessary, telephone. The Lab PM will describe the corrective action taken, or if necessary, provide options for the AI PM to consider. The AI PM will consult with the NPS COR prior to making a decision or will inform the NPS COR via email within 24 hours if a predetermined corrective action was taken.

### **7.3 Reconciliation with DQOs and Data Usability**

A draft data verification report will be provided to the NPS COR upon completion of the data verification process. This report will be a draft to the EE/CA report data assessment section. It will be available for review by the NPS within two months of the completion of the field work, estimated to be in late April or early May, 2021. The AI team will review the results with the NPS team, confirm data that are usable



for screening level assessments, and determine any data gaps that remain based on the DQOs provided in Section 4 of this SAP.





## 8. INVESTIGATION OUTPUTS

In accordance with the SOW (NPS 2020), an EE/CA consistent with the NPS EE/CA template will be prepared. This document will summarize historical results from the prior investigation (Terracon 2006), identify data gaps that have been filled, characterize the nature and extent of contamination, present the results of human health and ecological risk assessments, identify ARARs, and, if needed, develop preliminary, Site-specific, risk-based preliminary remediation goals (PRGs) and analyze an appropriate array of removal alternatives consistent with CERCLA and NCP.



## 9. REFERENCES

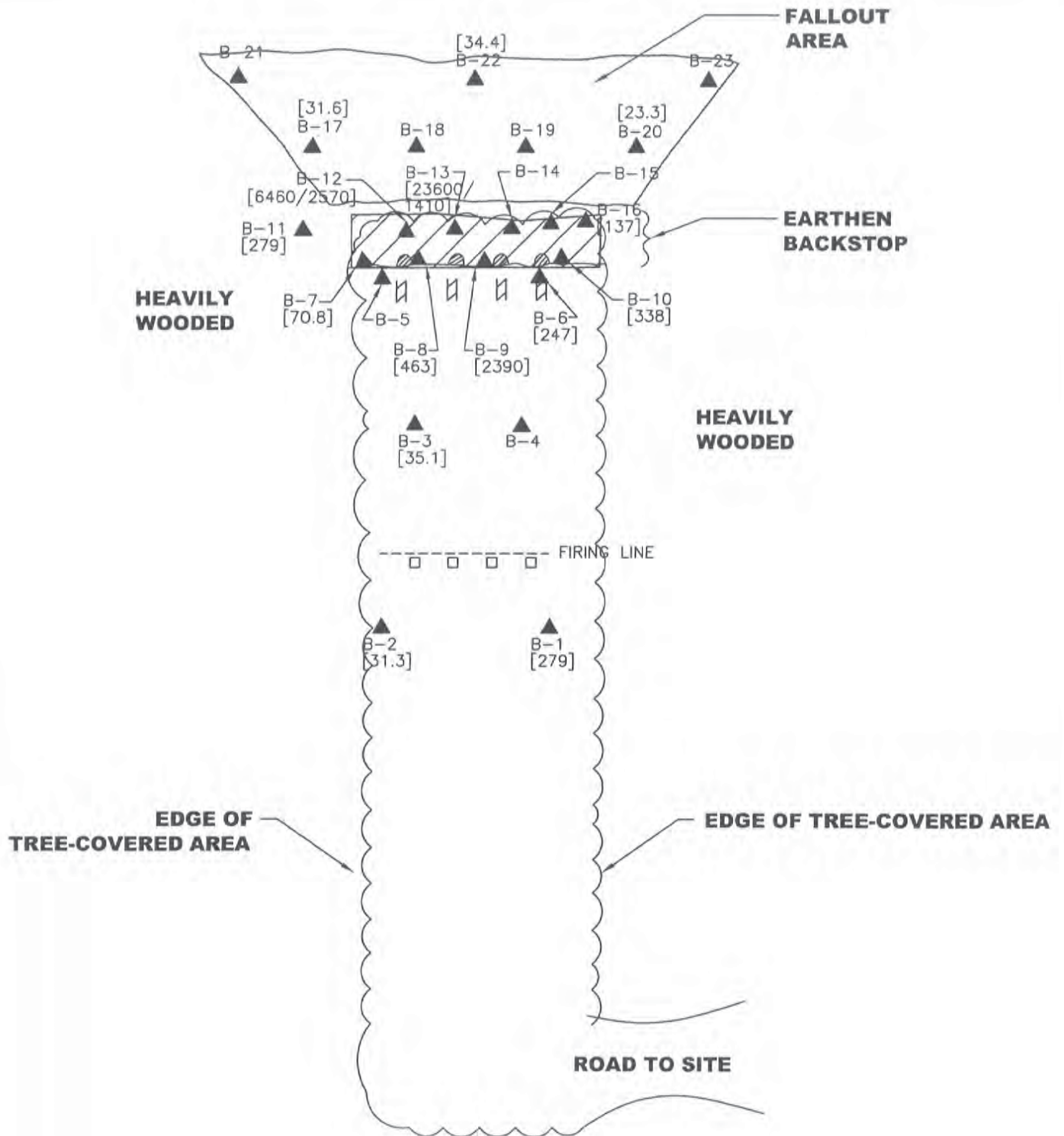
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## Appendix A – Site Assessment Report (Terracon 2006)







#### LEGEND

- PROPOSED EXCAVATION LIMIT
- TARGET
- WOODEN FIRING POST
- SAMPLE LOCATION, [CONCENTRATION OF LEAD AT 0-6"/6-12" IN MG/KG]



Approximate Scale (Feet)  
Scale factor=1:480



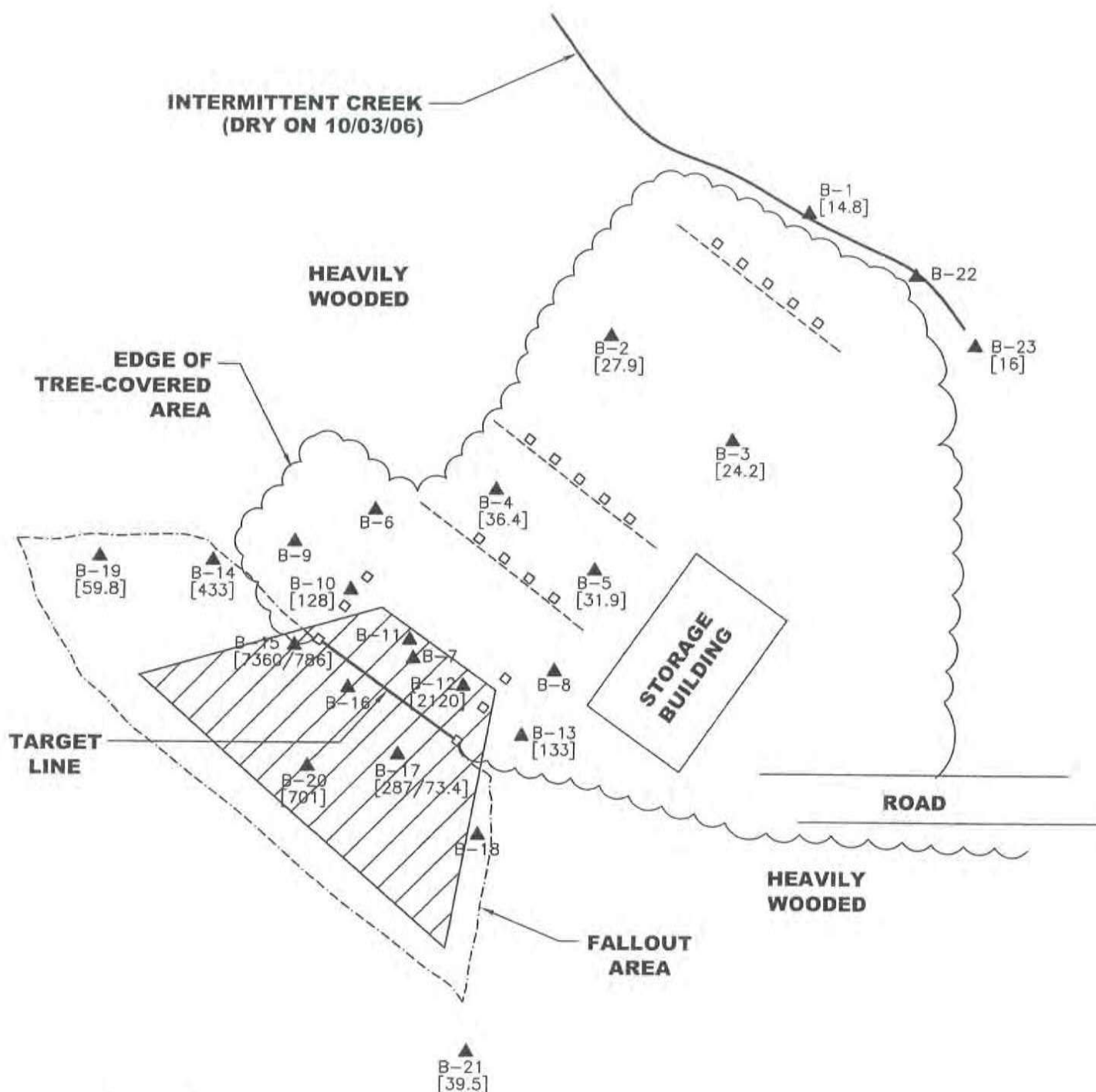
R3 PAI 11/16/06

#### SAMPLE LOCATION DIAGRAM OZARK SCENIC RIVERWAYS FIRING RANGES UPPER CURRENT FIRING RANGE AKERS, MISSOURI

Project Mngr:	JLD	Project No.	05067141
Designed By:	JLD	Scale:	AS SHOWN
Drawn By:	PAI	File No.	05067141C09
Checked By:	JLD	Date:	SEPT. 2006
Approved By:	RMB	Figure No.	4

**Terracon**

2211 S. 156th Circle  
Omaha, NE 68130



SAMPLE LOCATION DIAGRAM  
OZARK SCENIC RIVERWAYS FIRING RANGES  
BIG SPRING FIRING RANGE  
BIG SPRING, MISSOURI

Project Mngr: JLD  
Designed By: JLD  
Drawn By: PAI  
Checked By: JLD  
Approved By: RMB

**Terracon**  
2211 S. 156th Circle  
Omaha, NE 68130

Project No. 05067141  
Scale: AS SHOWN  
File No. 05067141C09  
Date: SEPT. 2006

Figure No.

TABLE 1

SUMMARY OF ANALYTICAL RESULTS  
UPPER CURRENT  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
VAN BUREN, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-1	0 to 6	1 unspent round			1488.1	13.19	279
B-2	0 to 6				1298.0		31.3
B-3	0 to 6		1 casing		1361.2		35.1
B-4	0 to 6		1 casing		1340.6		Not Analyzed
B-5	0 to 6				1013.8		Not Analyzed
B-6	0 to 6				1228.9		247
B-7	0 to 6				1044.2		70.8
B-8	0 to 6	1			1202.0	7.0	463
B-9	0 to 6	1			959.6	9.7	2,390
B-10	0 to 6	1	1		1453.8	7.64	338
B-11	0 to 6				1057.5		119
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800



TABLE 1

SUMMARY OF ANALYTICAL RESULTS  
UPPER CURRENT  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
VAN BUREN, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-12	0 to 6	13	46	14	1263.7	153.97	6,460
B-12	6 to 12	6	15		1209.5	62.99	2,570
B-13	0 to 6	18	15	5	1241.5	182.25	23,600
B-13	6 to 12				1287.3		1,410
B-14	0 to 6	20	33		1608.7	196.40	Not Analyzed
B-14	6 to 12				1333.7		Not Analyzed
B-15	0 to 6	20	37		1663.3	204.82	Not Analyzed
B-15	6 to 12				1392.6		Not Analyzed
B-16	0 to 6				1095.1		137
B-17	0 to 6				1222.8		31.6
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800



TABLE 1

SUMMARY OF ANALYTICAL RESULTS  
UPPER CURRENT  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
VAN BUREN, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-18	0 to 6				935.3		Not Analyzed
B-19	0 to 6				1323.2		Not Analyzed
B-20	0 to 6				1124.6		<b>23.3</b>
B-21	0 to 6				1086.2		Not Analyzed
B-22	0 to 6				1136.3		<b>34.4</b>
B-23	0 to 6				1016.1		Not Analyzed
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800

**NOTES:**

mg/kg indicates micrograms per kilogram which is equivalent to parts per million (ppm).

Bold indicates detection above the reporting limit.

<sup>(1)</sup> Indicates Missouri Department of Natural Resources (MoDNR) Target Levels for Lead, Non-residential land use soil (direct contact with soil), Missouri Risk-Based Corrective Action Technical Guidance, Appendix E, April 2006

<sup>(2)</sup> Indicates U.S. Environmental Protection Agency (USEPA) Preliminary Remediation Goals (PRG) for "Direct Contact Exposure Pathways", Industrial Soil

TABLE 2

SUMMARY OF ANALYTICAL RESULTS  
ALLEY SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
VAN BUREN, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-1	0 to 6				1629.7		10.9
B-2	0 to 6				1523.8		Not Analyzed
B-3	0 to 6				1418.3		Not Analyzed
B-4	0 to 6				1523.9		Not Analyzed
B-5	0 to 6				1176.9		Not Analyzed
B-6	0 to 6		15 casings		1498.6		37.9
B-7	0 to 6				1508.2		43.7
B-8	0 to 6				1279.3		Not Analyzed
B-9	0 to 6				1209.8		Not Analyzed
B-10	0 to 6		1		1182.6	0.80	59.0
B-11	0 to 6				1312.3		Not Analyzed
B-12	0 to 6				1091.4		71.5
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800



TABLE 2

SUMMARY OF ANALYTICAL RESULTS  
ALLEY SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
ALLEY SPRING, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-13	0 to 6	2	2	1	1190.3	17.96	3,770
B-14	0 to 6				1325.5		86.4
B-15	0 to 6				1076.8		Not Analyzed
B-16	0 to 6	6	8		1188.7	55.3	2,460
B-17	0 to 6				1198.7		43.2
B-18	0 to 6	3	1		1141.9	27.60	3,860
B-18	6 to 12	1			1120.1	9.68	488
B-19	0 to 6				1392.2		Not Analyzed
B-20	0 to 6				1409.0		Not Analyzed
B-21	0 to 6				1186.0		217
B-22	0 to 6				1268.3		26.1
B-23	0 to 6				1268.3		Not Analyzed
B-24	0 to 6				1182.5		31.6
B-25	0 to 6		1		1170.6	1.55	57.6
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800

TABLE 2

SUMMARY OF ANALYTICAL RESULTS  
ALLEY SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
ALLEY SPRING, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-26	0 to 6				1119.0		Not Analyzed
B-27	0 to 6				1210.0		50.6
B-28	0 to 6				1184.4		Not Analyzed
B-29	0 to 6				1079.7		Not Analyzed
B-30	0 to 6				1011.7		Not Analyzed
B-31	0 to 6				875.2		133
B-32	0 to 6	2	2		1190.6	13.9	816
B-33	0 to 6				1029.9		175
B-34	0 to 6	4	6 (aluminum casing)		1218.0	156.20	780
B-34	6 to 12				1312.5		258
B-35	0 to 6				1179.7		Not Analyzed
B-36	0 to 6				1253.2		Not Analyzed
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800



TABLE 2

SUMMARY OF ANALYTICAL RESULTS  
ALLEY SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
ALLEY SPRING, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-37	0 to 6				1179.9		23.4
B-38	0 to 6				1313.6		Not Analyzed
B-39	0 to 6				1008.7		21.5
B-40	0 to 6				1240.1		Not Analyzed
B-41	0 to 6				1272.5		Not Analyzed
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800

**NOTES:**

mg/kg indicates micrograms per kilogram which is equivalent to parts per million (ppm).

Bold indicates detection above the reporting limit.

<sup>(1)</sup> Indicates Missouri Department of Natural Resources (MoDNR) Target Levels for Lead, Non-residential land use soil (direct contact with soil), Missouri Risk-Based Corrective Action Technical Guidance, Appendix E, April 2006

<sup>(2)</sup> Indicates U.S. Environmental Protection Agency (USEPA) Preliminary Remediation Goals (PRG) for "Direct Contact Exposure Pathways", Industrial Soil

TABLE 3

SUMMARY OF ANALYTICAL RESULTS  
BIG SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
BIG SPRING, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-1	0 to 6				1180.9		14.8
B-2	0 to 6		1		1505.5	4.55	27.9
B-3	0 to 6				1708.4		24.2
B-4	0 to 6		2		1581.8	8.7	36.4
B-5	0 to 6				1886.5		31.9
B-6	0 to 6				1562.1		Not Analyzed
B-7	0 to 6				1759.3		Not Analyzed
B-8	0 to 6				1719.3		Not Analyzed
B-9	0 to 6				1886.3		Not Analyzed
B-10	0 to 6				1644.3		128
B-11	0 to 6		2 casings		1733.2		Not Analyzed
B-12	0 to 6	1	4		1733.9	9.19	2,120
B-13	0 to 6				1554.4		133
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)							800



TABLE 3  
(CONTINUED)

SUMMARY OF ANALYTICAL RESULTS  
BIG SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
BIG SPRING, MISSOURI

Terracon Project No. 05067141

Sample	Depth (inches)	Spent Lead Bullets	Lead & Brass Fragments	Lead Shot	Sample Mass (grams)	Uncleaned Lead Bullets Mass (grams)	Lead Concentration (mg/kg)
B-14	0 to 6				1424.1		433
B-15	0 to 6		4	9	1362.8	84.7	7,360
B-15	0 to 6				1574.6		786
B-16	0 to 6	10	5		1247.6	97.92	Not Analyzed
B-16	6 to 12				14622		Not Analyzed
B-17	0 to 6				1374.3		287
B-17	0 to 6		2		1397.0	5.16	73.4
B-18	0 to 6				1374.2		
B-18	6 to 12				1579.8		
B-19	0 to 6	2			1226.1	21.25	59.2
B-20	0 to 6	4	6		1432.6	39.98	701
B-21	0 to 6				1529.7		39.5
B-22	0 to 6				1462.8		Not Analyzed
B-23	0 to 6				1172.3		11.0
MoDNR Target Level (mg/kg) <sup>1</sup>							660
USEPA PRG <sup>2</sup> (mg/kg)				2			800

**Terracon**

TABLE 3  
(CONTINUED)

SUMMARY OF ANALYTICAL RESULTS  
BIG SPRING  
SMALL ARMS FIRING RANGE  
OZARK NATIONAL SCENIC RIVERWAYS  
BIG SPRING, MISSOURI

Terracon Project No. 05067141

**NOTES:**

mg/kg indicates micrograms per kilogram which is equivalent to parts per million (ppm).

Bold indicates detection above the reporting limit.

<sup>(1)</sup> Indicates Missouri Department of Natural Resources (MoDNR) Target Levels for Lead, Non-residential land use soil (direct contact with soil), Missouri Risk-Based Corrective Action Technical Guidance, Appendix E, April 2006

<sup>(2)</sup> Indicates U.S. Environmental Protection Agency (USEPA) Preliminary Remediation Goals (PRG) for "Direct Contact Exposure Pathways", Industrial Soil



November 10, 2006

Client:

TERRACON - OMAHA  
2211 South 156th Circle  
Omaha, NE 68130

Work Order: CPJ1747  
Project Name: Ozarks - Van Buren, MO  
Project Number: 05067141 - Upper Current

Attn: Janet Doan

Date Received: 10/31/06

An executed copy of the chain of custody is also included as an addendum to this report

If you have any questions relating to this analytical report, please contact your Laboratory Project Manager at 1-(800)750-2401

SAMPLE IDENTIFICATION	LAB NUMBER	COLLECTION DATE AND TIME
B-1	CPJ1747-01	10/04/06 13:15
B-2	CPJ1747-02	10/04/06 13:20
B-3	CPJ1747-03	10/04/06 13:25
B-6	CPJ1747-04	10/04/06 13:40
B-7	CPJ1747-05	10/04/06 13:49
B-8	CPJ1747-06	10/04/06 13:53
B-9	CPJ1747-07	10/04/06 14:02
B-10	CPJ1747-08	10/04/06 14:10
B-11	CPJ1747-09	10/04/06 14:25
B-12 0-6 in.	CPJ1747-10	10/04/06 14:35
B-12 6-12 in.	CPJ1747-11	10/04/06 14:35
B-13 0-6 in.	CPJ1747-12	10/04/06 14:45
B-13 6-12 in.	CPJ1747-13	10/04/06 14:45
B-16	CPJ1747-14	10/04/06 15:20
B-17	CPJ1747-15	10/04/06 15:25
B-20	CPJ1747-16	10/04/06 15:45
B-22	CPJ1747-17	10/04/06 15:50

**Samples were received into laboratory on ice.**

Most environmental analytical testing methods require a sample temperature of 4 degrees C +/- 2 degrees C for preservation of the sample constituents prior to analysis. If sample temperatures are outside of this temperature range at the time of sample receipt, results may be impacted. Please refer to the Temperature and Sample Receipt form that is included with this report for additional information regarding the condition of samples at the time of receipt by the laboratory.

The reported results were obtained in compliance with the 2003 NELAC standards unless otherwise noted.

*Reproduction of this analytical report is permitted only in its entirety. This report shall not be reproduced except in full without the written approval of the laboratory.*

*TestAmerica Analytical Testing Corporation certifies that the analytical results contained herein apply only to the specific sample analyzed.*

# TestAmerica

ANALYTICAL TESTING CORPORATION

704 Enterprise Drive Cedar Falls, IA 50613 • 800-750-2401 • Fax 319-277-2425

TERRACON - OMAHA  
2211 South 156th Circle  
Omaha, NE 68130  
Janet Doan

Work Order: CPJ1747  
Project: Ozarks - Van Buren, MO  
Project Number: 05067141 - Upper Current

Received: 10/31/06  
Reported: 11/10/06 15:16

Approved By:



TestAmerica - Cedar Falls, IA  
Linda Cmelik  
Project Coordinator

TERRACON - OMAHA  
2211 South 156th Circle  
Omaha, NE 68130  
Janet Doan

Work Order: CPJ1747  
Project: Ozarks - Van Buren, MO  
Project Number: 05067141 - Upper Current

Received: 10/31/06  
Reported: 11/10/06 15:16

## ANALYTICAL REPORT

Analyte	Sample Result	Data Qualifiers	Units	Quan. Limit	Dilution Factor	Date Analyzed	Seq/ Analyst Batch	Method
Sample ID: CPJ1747-01 (B-1 - Soil)					Sampled: 10/04/06 13:15		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	76.2	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	279		mg/kg dry	1.30	0.994	11/02/06 20:37	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-02 (B-2 - Soil)					Sampled: 10/04/06 13:20		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	89.0	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	31.3		mg/kg dry	1.10	0.982	11/02/06 20:42	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-03 (B-3 - Soil)					Sampled: 10/04/06 13:25		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	84.9	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	35.1		mg/kg dry	1.16	0.988	11/02/06 21:08	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-04 (B-6 - Soil)					Sampled: 10/04/06 13:40		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	91.9	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	247		mg/kg dry	1.07	0.984	11/02/06 21:12	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-05 (B-7 - Soil)					Sampled: 10/04/06 13:49		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	87.0	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	70.8		mg/kg dry	1.12	0.978	11/02/06 21:17	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-06 (B-8 - Soil)					Sampled: 10/04/06 13:53		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	90.6	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	463		mg/kg dry	1.09	0.986	11/02/06 21:22	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-07 (B-9 - Soil)					Sampled: 10/04/06 14:02		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	88.8	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	2390		mg/kg dry	11.1	9.86	11/03/06 10:03	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-08 (B-10 - Soil)					Sampled: 10/04/06 14:10		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	93.0	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G
Total Metals by EPA Method 6010B								
Lead	338		mg/kg dry	1.03	0.958	11/02/06 21:31	jwd 6110262	SW846 6010B
Sample ID: CPJ1747-09 (B-11 - Soil)					Sampled: 10/04/06 14:25		Recvd: 10/31/06 09:15	
General Chemistry Parameters								
Total Solids	81.2	H3	%	NA	1	11/01/06 22:51	JHS 6110240	SM 2540G



TERRACON - OMAHA  
2211 South 156th Circle  
Omaha, NE 68130  
Janet Doan

Work Order: CPJ1747  
Project: Ozarks - Van Buren, MO  
Project Number: 05067141 - Upper Current

Received: 10/31/06  
Reported: 11/10/06 15:16

## ANALYTICAL REPORT

Analyte	Sample Result	Data Qualifiers	Units	Quan. Limit	Dilution Factor	Date Analyzed	Analyst	Seq/ Batch	Method
Sample ID: CPJ1747-09 (B-11 - Soil) - cont.					Sampled: 10/04/06 14:25		Recvd: 10/31/06 09:15		
Total Metals by EPA Method 6010B									
Lead	119		mg/kg dry	1.20	0.973	11/02/06 21:35	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-10 (B-12 0-6 in. - Soil)					Sampled: 10/04/06 14:35		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	92.5	H3	%	NA	1	11/01/06 22:51	JHS	6110240	SM 2540G
Total Metals by EPA Method 6010B									
Lead	6460		mg/kg dry	10.7	9.92	11/03/06 10:06	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-11 (B-12 6-12 in. - Soil)					Sampled: 10/04/06 14:35		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	91.8	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	2570		mg/kg dry	10.7	9.86	11/03/06 10:10	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-12 (B-13 0-6 in. - Soil)					Sampled: 10/04/06 14:45		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	92.6	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	23600		mg/kg dry	108	99.8	11/03/06 10:20	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-13 (B-13 6-12 in. - Soil)					Sampled: 10/04/06 14:45		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	91.6	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	1410		mg/kg dry	1.05	0.962	11/02/06 22:10	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-14 (B-16 - Soil)					Sampled: 10/04/06 15:20		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	93.3	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	137		mg/kg dry	1.07	1	11/02/06 22:15	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-15 (B-17 - Soil)					Sampled: 10/04/06 15:25		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	90.2	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	31.6		mg/kg dry	1.07	0.965	11/02/06 22:19	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-16 (B-20 - Soil)					Sampled: 10/04/06 15:45		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	91.0	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	23.3		mg/kg dry	1.09	0.996	11/02/06 22:24	jwd	6110262	SW846 6010B
Sample ID: CPJ1747-17 (B-22 - Soil)					Sampled: 10/04/06 15:50		Recvd: 10/31/06 09:15		
General Chemistry Parameters									
Total Solids	77.4	H3	%	NA	1	11/02/06 00:53	JHS	6110241	SM 2540G
Total Metals by EPA Method 6010B									
Lead	34.4		mg/kg dry	1.28	0.988	11/02/06 22:29	jwd	6110262	SW846 6010B



TERRACON - OMAHA  
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Omaha, NE 68130  
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Work Order: CPJ1747  
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Received: 10/31/06  
Reported: 11/10/06 15:16

## LABORATORY BLANK QC DATA

Analyte	Seq/ Batch	Source Spike Result Level	Units	MDL	MRL	Result	Dup Result	% REC	Dup %REC	REC Limits	RPD RPD	Limit	Q
<b>General Chemistry Parameters</b>													
Total Solids	6110240		%	N/A	N/A	-0.000610							
Total Solids	6110241		%	N/A	N/A	-0.000200							
<b>Total Metals by EPA Method 6010B</b>													
Lead	6110262		mg/kg wet	N/A	0.978	<0.978							

TERRACON - OMAHA  
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## LABORATORY DUPLICATE QC DATA

Analyte	Seq/ Batch	Source Spike Result Level	Units	MDL	MRL	Result	% REC	Dup %REC	% REC Limits	RPD RPD	RPD Limit	Q
<b>General Chemistry Parameters</b>												
<b>QC Source Sample: CPJ1747-10</b>												
Total Solids	6110240	92.5	%	N/A	N/A	93.3				1	20	
<b>QC Source Sample: NPK0110-03</b>												
Total Solids	6110241	68.4	%	N/A	N/A	69.6				2	20	

TERRACON - OMAHA  
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Received: 10/31/06  
Reported: 11/10/06 15:16

## LCS/LCS DUPLICATE QC DATA

Analyte	Seq/ Batch	Source Spike Result Level	Units	MDL	MRL	Result	Dup Result	% REC	Dup %REC	% REC Limits	RPD RPD	Limit	Q
<b>General Chemistry Parameters</b>													
Total Solids	6110240	0.0100	%	N/A	N/A	0.00976		98		90-110			
Total Solids	6110241	0.0100	%	N/A	N/A	0.00915		92		90-110			
<b>Total Metals by EPA Method 6010B</b>													
Lead	6110262	100	mg/kg wet	N/A	1.00	99.9		100		80-120			

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Received: 10/31/06  
Reported: 11/10/06 15:16

## MATRIX SPIKE/MATRIX SPIKE DUPLICATE QC DATA

Analyte	Seq/ Batch	Source Spike Result Level	Units	MDL	MRL	Dup Result	% REC	Dup Result	% REC	REC Limits	RPD	RPD Limit	Q
Total Metals by EPA Method 6010B													
QC Source Sample: NPK0080-04													
Lead	6110262	8.69	101	mg/kg wet	N/A	1.01	106	103	96	95	75-125	3	20



TERRACON - OMAHA  
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Omaha, NE 68130  
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Project: Ozarks - Van Buren, MO  
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Received: 10/31/06  
Reported: 11/10/06 15:16

## CERTIFICATION SUMMARY

### Subcontracted Laboratories

TestAmerica Analytical Testing Corp.- Nashville NELAC Cert #87358, Illinois Cert #001366, Iowa Cert #131, Kansas Cert #E-10229, Minnesota Cert #047-999-345, Wisconsin Cert #998020436

2960 Foster Creighton Dr. - Nashville, TN 37204

Method Performed: SM 2540G

Samples: CPJ1747-01, CPJ1747-02, CPJ1747-03, CPJ1747-04, CPJ1747-05, CPJ1747-06, CPJ1747-07, CPJ1747-08, CPJ1747-09, CPJ1747-10, CPJ1747-11, CPJ1747-12, CPJ1747-13, CPJ1747-14, CPJ1747-15, CPJ1747-16, CPJ1747-17

Method Performed: SW846 6010B

Samples: CPJ1747-01, CPJ1747-02, CPJ1747-03, CPJ1747-04, CPJ1747-05, CPJ1747-06, CPJ1747-07, CPJ1747-08, CPJ1747-09, CPJ1747-10, CPJ1747-11, CPJ1747-12, CPJ1747-13, CPJ1747-14, CPJ1747-15, CPJ1747-16, CPJ1747-17

*Any abnormalities or departures from sample acceptance policy shall be documented on the 'Sample Receipt and Temperature Log Form' and 'Sample Non-conformance Form' (if applicable) included with this report.*

*For information concerning certifications of this facility or another TestAmerica facility, please visit our website at [www.TestAmericaInc.com](http://www.TestAmericaInc.com)*

*Samples collected by TestAmerica Field Services personnel are noted on the Chain of Custody (COC) and are sampled in accordance with TA-CF SOP CF09-01.*

## DATA QUALIFIERS AND DEFINITIONS

H3 Sample was received and analyzed past holding time

## ADDITIONAL COMMENTS

Results are reported on a wet weight basis unless otherwise noted.

To assist us in using the proper analytical methods,  
is this work being conducted for regulatory purposes?  
Compliance Monitoring

Client Name: Terracon Client #: \_\_\_\_\_  
Address: 2211 S. 156th Circle  
City/State/Zip Code: Omaha NE 68130  
Project Manager: Janet Dorn  
Telephone Number: 402-330-2202 Fax: 402-330-7601  
Sampler Name: (Print Name) Michael Henry  
Sampler Signature: Michael Henry  
Email Address: ideal@terracon.com

Client #:

Address: 2211 S. 156th Circle  
City/State/Zip Code: Omaha NE 68130  
Project Manager: Janet Donah  
Telephone Number: 402-330-2202 Fax: 402-330-7601  
Sampler Name: (Print Name) Michael Henry  
Sampler Signature: Michael Henry

Email Address: illean@terracon.com

Project Name:  
Project #:  
Site/Location ID:  
Report To:  
Invoice To:  
Quote #:

PO#

TAT		Date Needed:		Date Sampled		Time Sampled		Field Filtered		Matrix		Preservation & # of Containers		Analyze For:		QC Deliverables	
<input type="checkbox"/> Standard <input type="checkbox"/> Rush (surcharges may apply)								G = Grab, C = Composite Field Filtered		SL - Sludge DW - Drinking Water GW - Groundwater S - Soil/Solid WW - Wastewater Specify Other		HNO <sub>3</sub> HCl NaOH H <sub>2</sub> SO <sub>4</sub> Methanol None Other (Specify)		Total Lead (6010)		None Level 2 (Batch QC) Level 3 Level 4 Other:	
SAMPLE ID		Fax Results: Y N		Email Results: Y N												REMARKS	
B-1						10/4/06 1:15		G		S		X					
B-2						1:20											
B-3						1:25											
B-4						1:40											
B-7						1:49											
B-8						1:53											
B-9						2:02											
B-10						2:10											
B-11						2:25											
Special Instructions:																	
LABORATORY COMMENTS:																	
Relinquished By: <i>Robert Dean</i>		Date: 10/3/08		Time: 3:45		Received By: <i>Sam M. Williams</i>		Date: 10/3/08		Time: 9:15							
Relinquished By:		Date:		Time:		Received By:		Date:		Time:							
Relinquished By:		Date:		Time:		Received By:		Date:		Time:							



Cedar Falls Division  
704 Enterprise Drive  
Cedar Falls, IA 50613

Phone 319-277-2401 or 800-750-2401  
Fax 319-277-2425

**To assist us in using the proper analytical methods,  
is this work being conducted for regulatory purposes?**  
Compliance Monitoring

Client Name: Terracon Client #: \_\_\_\_\_  
Address: 2211 S. 156th Circle  
City/State/Zip Code: Omaha NE 68130  
Project Manager: Tawet-Tean  
Telephone Number: 402-330-2202 Fax: 402-330-7606  
Sampler Name: (Print Name) Michael Henry  
Sampler Signature: Michael Henry  
Email Address: jldean@terracon.com

Project Name: Ozark - Upper Current  
Project #: 05067141 Task 2  
Site/Location ID: Van Buren State: MO  
Report To: Janet Doan  
Invoice To: Janet Doan  
Quote #: PO#:

TAT		Date Needed:		Date Sampled		Time Sampled		G = Grab, C = Composite		Field Filtered		Matrix		Preservation & # of Containers		Analyze For:		QC Deliverables	
Standard		Rush (surcharges may apply)										SL - Sludge DW - Drinking Water GW - Groundwater S - Soil/Solid WW - Wastewater Specify, Other		HNO <sub>3</sub> HCl NaOH H <sub>2</sub> SO <sub>4</sub> Methanol None Other (Specify)		(6010) Lead Total Lead		None Level 2 (Batch QC) Level 3 Level 4 Other:	
B-12 (0-6")		Y N		10/14/06		2:35		G				S		None					
B-12 (6-12")		Y N		2:35		G						S		None					
B-13 (0-6")		Y N		2:45		G								None					
B-13 (6-12")		Y N		2:45		G								None					
B-16		Y N		3:20		G								None					
B-17		Y N		3:25		G								None					
B-20		Y N		3:45		G								None					
B-22		Y N		3:50		G								None					

Special Instructions:

LABORATORY COMMENTS:

Relinquished By: <i>John Doe</i>	Date: 10/30/06	Time: 3:45	Received By: <i>Sam Johnson</i>	Date: 10/31/06	Time: 9:15
Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Relinquished By:	Date:	Time:	Received By:	Date:	Time:



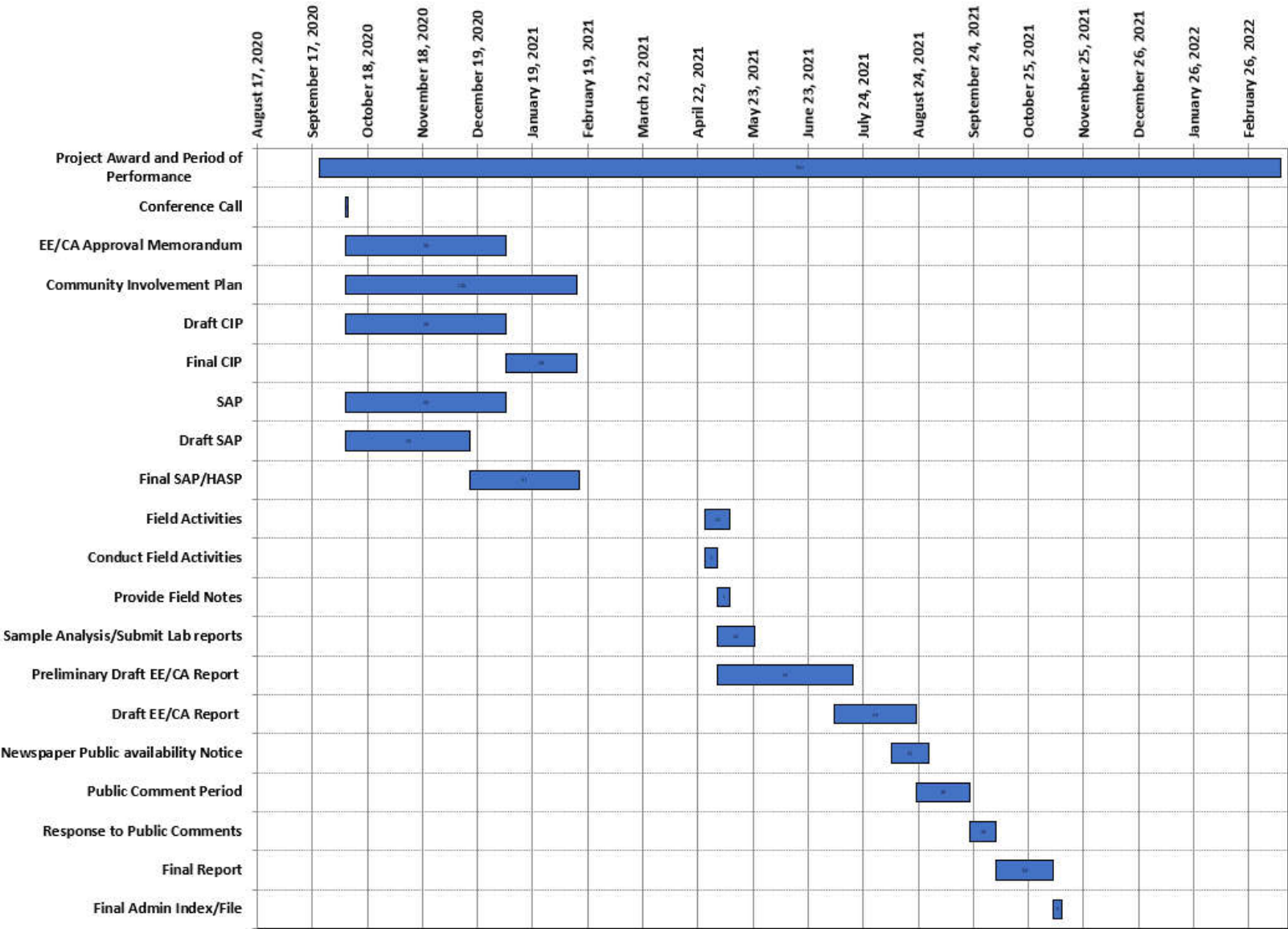
## **Appendix B – AI Project Schedule**





AppB-1. Comprehensive Schedule  
Upper Current and Big Springs SAFRs, Ozark National Scenic Riverways

WBS	Task	Start Date	Duration	End Date	Comments
1.1	Project Award and Period of Performance	Monday, September 21, 2020	541	Wednesday, March 16, 2022	Set by contract.
1.2	Conference Call	Tuesday, October 6, 2020	1	Wednesday, October 7, 2020	No later than 10 calendar days after award.
1.3	EE/CA Approval Memorandum	Tuesday, October 6, 2020	90	Monday, January 4, 2021	Ask for end date
1.4	Community Involvement Plan	Tuesday, October 6, 2020	130	Saturday, February 13, 2021	Assumes draft-20 days, review-20 days, final-10 days.
1.4.1	Draft CIP	Tuesday, October 6, 2020	90	Monday, January 4, 2021	Assumes 90 days for draft
1.4.2	Final CIP	Monday, January 4, 2021	40	Saturday, February 13, 2021	Assumes 30 days for review, 10 days for finalization
1.5	SAP	Tuesday, October 6, 2020	90	Monday, January 4, 2021	
1.5.1	Draft SAP	Tuesday, October 6, 2020	70	Tuesday, December 15, 2020	Assumes 70 days for draft
1.5.2	Final SAP/HASP	Tuesday, December 15, 2020	61	Sunday, February 14, 2021	Assumes 30 days review, 10 days to resubmit
2.1	Field Activities	Monday, April 26, 2021	14	Monday, May 10, 2021	Field Activities to start in Spring 2021
2.1.1	Conduct Field Activities	Monday, April 26, 2021	7	Monday, May 3, 2021	Assume 1 week for fieldwork.
2.1.2	Provide Field Notes	Monday, May 3, 2021	7	Monday, May 10, 2021	Field notes to be provided 1 week after the completion of field effort
2.1.3	Sample Analysis/Submit Lab reports	Monday, May 3, 2021	21	Monday, May 24, 2021	Laboratory reports provided 3 weeks after completion of fieldwork
3.1	Preliminary Draft EE/CA Report	Monday, May 3, 2021	76	Thursday, July 8, 2021	Preliminary Draft EE/CA Report 76 days after completion of field work.
3.2	Draft EE/CA Report	Thursday, July 8, 2021	46	Monday, August 23, 2021	Assumes 30 days for comments, 15 days to provide RTCs and Draft Report for public review.
3.3	Newspaper Public availability Notice	Monday, August 9, 2021	21	Monday, August 30, 2021	Assumes notice of availability will be 2 weeks before draft document is available
3.4	Public Comment Period	Monday, August 23, 2021	30	Wednesday, September 22, 2021	Assumes 30 day public comment period
3.5	Response to Public Comments	Wednesday, September 22, 2021	15	Thursday, October 7, 2021	Assumes 15 days to compile and provide response to public comments
3.6	Final Report	Thursday, October 7, 2021	32	Monday, November 8, 2021	Assumes 30 days to Finalize the EE/CA Report
3.7	Final Admin Index/File	Monday, November 8, 2021	5	Saturday, November 13, 2021	Assumes 5 days to provide the final Administrative Record files and index.





## Appendix C – AI Standard Operating Procedures

## SECTION 1 FIELD OPERATIONS

### 1.1 GEOLOGIC STANDARDS SOP (AI – W FO-01)

The lithologic descriptions for consolidated materials (igneous, metamorphic, and sedimentary rocks) shall follow the standard professional nomenclature (Tennissen, 1983), with special attention given to describing fractures, vugs, solution cavities and their fillings or coatings, and any other characteristics affecting permeability. Colors shall be designated by the Munsell Color System.

The lithologic descriptions for unconsolidated materials (soils [engineering usage] or deposits) shall use the name of the predominant particle size (e.g., silt, fine sand, etc.). The dimensions of the predominant and secondary sizes shall be recorded using the metric system. The grain size and name of the deposit shall be accompanied by the predominant mineral content, accessory minerals, color, particle angularity, and any other characteristics. The clastic deposit descriptions shall include, as a supplement, symbols of the Unified Soil Classification System (USCS). The color descriptions shall be designated by the Munsell Color System.

The scales for maps, cross sections, or 3-D diagrams shall be selected in accordance with the geologic and hydrologic complexity of the area and the purposes of the illustrations. Geophysical logs shall be run at a constant vertical scale of 1 inch equals 20 feet. When geophysical logs are superimposed on geologic logs, cross sections, or 3-D diagrams, the scales shall be the same. If defining geological conditions requires other scales, additional logs at those scales shall be provided.

For orientation, the cross sections shall show the Northern end on the viewer's right. If the line of cross section is predominantly East-West, the Eastern end is on the right. Maps shall be oriented with North toward the top, unless the shape of the area dictates otherwise. Indicate orientation with a North arrow.

The USCS is a soil classification system used engineering and geology to describe the texture and grain size of a soil. The classification system can be applied to most unconsolidated materials, and is represented by a two-letter symbol. Each letter is described below (with the exception of Pt [peat]):

Letter	G	S	M	O
Definition	gravel	sand	silt	organic

If the soil has 5–12% by weight of fines passing a #200 sieve ( $5\% < P_{\#200} < 12\%$ ), both grain size distribution and plasticity have a significant effect on the engineering properties of the soil, and dual notation may be used for the group symbol. For example, GW-GM corresponds to "well-graded gravel with silt."

If the soil has more than 15% by weight retained on a #4 sieve ( $R_{\#4} > 15\%$ ), there is a significant amount of gravel, and the suffix "with gravel" may be added to the group name, but the group symbol does not change. For example, SP-SM could refer to "poorly graded SAND with silt" or "poorly graded SAND with silt and gravel."



### 1.1.1 Symbol Chart

Major Divisions			Group Symbol	Group Name
Coarse grained soils more than 50% retained on or above No.200 (0.075 mm) sieve	gravel > 50% of coarse fraction retained on No. 4 (4.75 mm) sieve	clean gravel <5% smaller than #200 Sieve	GW	well-graded gravel, fine to coarse gravel
			GP	poorly graded gravel
		gravel with >12% fines	GM	Silty gravel
			GC	clayey gravel
	sand ≥ 50% of coarse fraction passes No.4 sieve	clean sand	SW	well-graded sand, fine to coarse sand
			SP	poorly graded sand
		sand with >12% fines	SM	silty sand
			SC	clayey sand
Fine grained soils 50% or more passing the No.200 sieve	silt and clay liquid limit < 50	inorganic	ML	silt
			CL	clay of low plasticity, lean clay
		organic	OL	organic silt, organic clay
	silt and clay liquid limit ≥ 50	inorganic	MH	silt of high plasticity, elastic silt
			CH	clay of high plasticity, fat clay
		organic	OH	organic clay, organic silt
Highly organic soils			Pt	peat

## 1.2 SITE RECONNAISSANCE, PREPARATION AND RESTORATION SOP (AI-W FO-02)

The Contractor shall conduct a site reconnaissance of the field sites prior to the start of the field investigations. The primary objectives of conducting a site reconnaissance are to recommend possible changes in the technical approach, and to allow for adequate review of any such changes. During the site reconnaissance, the Contractor shall perform the following tasks:

- Review pertinent documents;
- Interview personnel who may be knowledgeable about environmental conditions in the project area;
- Conduct multiple site walks to evaluate monitoring and sampling locations;
- Verify and mark proposed sampling locations;
- Evaluate site accessibility and security;
- Designate field office sites;
- Identify potentially contaminated areas (i.e., discolored soils, stressed vegetation), particularly those that may require emergency response; and
- Document and evaluate site reconnaissance observations and update site maps.

A geophysical survey shall be conducted for the presence of underground utilities using electromagnetic methods in areas designated for intrusive sampling. Utility locations are determined using existing utility maps, and in the field, are verified using a hand-held magnetometer or utility probe. Vehicle access routes to sampling locations shall be determined prior to any field activity. Prior to any intrusive fieldwork, the Contractor Project Manager (PM) shall coordinate with the base Facilities Remedial Project Manager (RPM) to insure that the most current base procedures for obtaining dig permits and utility clearances are followed. In addition, any fieldwork activities along active flight line corridors or

other controlled access areas will require close coordination with the RPM to obtain access approval before fieldwork is initiated.

A centralized decontamination area shall be provided for drilling rigs and equipment. The decontamination area shall be large enough to allow storage of cleaned equipment and materials prior to use, as well as to stage drums of decontamination waste. Smaller decontamination areas for personnel and portable equipment shall be provided, as necessary. A designated location shall be provided for the field office if necessary.

Each work site or sampling location shall be returned to its original condition when possible. Efforts shall be made to minimize impacts to work sites and sampling locations, particularly those in or near sensitive environments such as wetlands. Following the completion of work at a site, all drums, trash, and other waste shall be removed and disposed of properly. Decontamination and/or purge water and soil cuttings shall be transported to the designated locations, as described in Section 1.17 and the site-specific WORKPLAN.

Site restoration shall be coordinated with the Facilities RPM and the field team leader (FTL) to ensure that site restoration is conducted according to facility requirements. A site restoration contractor shall be retained by the Contractor where necessary to restore areas following heavy equipment usage or drilling activity.

### **1.3 UTILITIES LOCATION SOP (AI – W FO-03)**

Prior to any intrusive work, the Contractor shall work with the Facilities RPM to follow project –specific requirements and current Facility requirements for Dig Permits. Careful planning plays an important role and is the first step in utility damage prevention efforts. Health and Safety Plans for projects that include subsurface drilling or excavation shall include a Site-Specific Hazard Analysis for, but not limited to, safety and provisions for utility line damage prevention. The Drilling Subcontractor and the Contractor shall determine the actual location of all known utilities through careful review of the civil/utility drawings before drilling in areas where the safety of existing utilities may be compromised, and shall evaluate aboveground features such as, but not limited to, manhole investigation (lifting manhole covers), valve boxes and pipe and cable risers which indicate the location of underground utilities prior to excavation/drilling. In general, a qualified utilities location subcontractor shall be contracted to conduct this work.

It is mandatory that the pertinent utility Location Service Center be notified and that each location of intrusive work is cleared by the proper authority prior to conducting subsurface work. The service center must be called at least three days before excavation and clearance is good for 15 days following clearance. All utilities in a work area shall be marked prior to subsurface work, using of stakes, flags, paint or other clearly identifiable materials to show the field location of underground utilities in accordance with current color code standards such as the American Public Works Association. This process is initiated with a call to the Local Utility Locate Service Center, requesting locates be made and providing the exact location of the drill site.

Following the location and marking of known or suspected utilities and excavation locations are located away from known underground utilities; initial digging shall be conducted using a shovel or backhoe at a careful rate with observers evaluating the first 4 feet of excavation depth closely for signs of man-made material such as conduit, metal, or concrete. This is done to uncover any potential utilities not identified by other means, especially where drilling or excavating is required within 3 feet of known or suspected utilities, or if exact locations are not known.

If there is any indication that the drill may have encountered a man-made obstruction within the first 10 feet below ground surface (bgs), operations shall stop immediately, and the excavation shall be

investigated prior to proceeding with subsurface work to determine whether that an unknown or improperly located utility may have been encountered.

If an existing utility is damaged, the Contractor Project Manager shall be notified immediately. The onsite Field Manager shall document the damage and pertinent circumstances surrounding the incident as soon as possible (in writing and with photographs or video), and a detailed incident/accident report shall be prepared.

#### **1.4 LITHOLOGIC LOGGING SOP (AI – W FO-07)**

The lithology in all boreholes shall be logged. The Drill Log form, (Appendix A), shall be used for recording the lithologic logging information. Information on the boring log sheet includes the borehole location; drilling information; sampling information such as sample intervals, recovery, and blow counts; and sample description information.

Unconsolidated samples for lithologic description shall be obtained at each change in lithology or every five (5) foot interval, whichever is less or as specifically stated in the SOW. Lithologic descriptions of unconsolidated materials encountered in the boreholes shall generally be described in accordance with American Society for Testing and Materials (ASTM) D-2488-90 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) (ASTM, 1990). Descriptive information to be recorded in the field shall include: (1) identification of the predominant particles size and range of particle sizes, (2) percent of gravel, sand, fines, or all three, (3) description of grading and sorting of coarse particles, (4) particle angularity and shape, and (5) maximum particle size or dimension.

Plasticity of fines description include: (1) color using Munsell Color System, (2) moisture (dry, wet, or moist), (3) consistency of fine grained soils, (4) structure of consolidated materials, and (4) cementation (weak, moderate, or strong).

Identification of the Unified Soil Classification System (USCS) group symbol shall be used. Additional information to be recorded includes the depth to the water table, caving or sloughing of the borehole, changes in drilling rate, depths of laboratory samples, presence of organic materials, presence of fractures or voids in consolidated materials, and other noteworthy observations or conditions, such as the locations of geologic boundaries.

Lithologic descriptions of consolidated materials encountered in the boreholes shall generally be described in accordance with Section 1.1. Consolidated samples for lithologic description shall be obtained at each change in lithology or at five-foot intervals, whichever is less, or as specified in the SOW. All samples shall be monitored with an organic vapor monitor (e.g., PID, organic vapor analyzer [OVA]). The samples shall be handled in such a way as to minimize the loss of volatiles, as described in Section 2.4. Cuttings shall be examined for their hazardous characteristics. Materials suspected to be hazardous because of abnormal color, odor, or organic vapor monitor readings shall be containerized in conformance with the Resource Conservation and Recovery Act (RCRA) and the state and local requirements. Rock cores shall be stored in standard core boxes, and missing sections of core shall be replaced with spacers.

Lithologic descriptions of consolidated materials shall follow the specifications in Section 1.1.

## **1.5 EQUIPMENT DECONTAMINATION SOP (AI – W FO – 16)**

All field equipment that may directly or indirectly contact samples shall be decontaminated in a designated decontamination station. A decontamination station shall be established at each field site prior to initiating intrusive field activities.

### **1.6.1 Instrument and Reusable Sampling Equipment Decontamination**

Instruments that contact water, such as an electronic water level indicator, shall be decontaminated following the procedures for sampling equipment described below. Instruments that are sensitive to soap and solvents, such as the pH meter, shall be rinsed with potable and deionized water. The probes shall be cleaned daily and stored overnight according to the manufacturer's recommended procedures.

All reusable field equipment used to collect, handle, or measure samples shall be decontaminated before coming into contact with any soil or groundwater samples. The decontamination procedure must match the degree of contamination of the sampling tool. For example, steam cleaning shall be necessary to remove dirt from auger flights and to prepare well screens and riser pipe for installation into the borehole. Brushes and soap may be required to remove dirt from split-spoon samplers. Sampling equipment that comes into contact with oil and grease shall be cleaned with methanol and hexane to remove any oily material. Clean, disposable gloves shall be worn during and after decontamination so that equipment shall not be re-contaminated.

Equipment decontamination at the Site will be limited to the stainless-steel sampling hand-trowel or spoons used to collect the soil/tailings and sediment samples, and the vessel used to collect water quality measurements during surface water sampling. This equipment will be sprayed with an Alconox® detergent solution and de-ionized water rinse between sampling units. Any other equipment that is visibly contaminated shall require decontamination.

Decontamination of sensitive instrumentation which requires disassembly of the instrument shall follow the procedures described by the manufacturer in the instrument manual.

General decontamination procedures for sampling and drilling devices such as spoon samplers, hand-operated soil probes, shovels, and other equipment that can be hand manipulated are as follows.

- Scrub equipment with a solution of potable water and a laboratory grade phosphate-free detergent (e.g., Alconox) to remove all dirt from sampling or drilling item;
- Rinse sampling item thoroughly with potable water to remove residual dirt and rewash, if necessary;
- If glass sampling equipment is used and metals are part of the analysis program, rinse the interior/exterior surfaces of the glass equipment that will come in contact with the sample matrix with 10 percent nitric acid (HNO<sub>3</sub>) solution.;
- Rinse item with analyte-free water, and
- Rinse a final time with analyte free water. This last rinse water can be captured and containerized, labeled wrapped and cooled to 4° C in a cooler as the Equipment Blank for the day.

## **1.7 WASTE HANDLING SOP (AI – W FO – 17)**

The Contractor shall prepare an updated Waste Management and Minimization Plan (WMMP) for the required activity. The WMMP shall be included as part of the site-specific WORK PLAN and details waste handling procedures that shall be implemented during the field investigation. A Waste Inventory Tracking Log (Appendix A) shall be kept during sampling events. Non investigative waste, such as litter and household garbage, shall be collected on an as-needed basis to maintain each site in a clean and orderly manner. This waste shall be containerized and transported to the designated sanitary landfill or



collection bin. Acceptable containers shall be sealed boxes or plastic garbage bags.

As designated in the SOW, soil cuttings and decontamination water will be disposed in the soil boring from which it originated.

### **1.8 SURVEYING SOP (AI – W FO – 18)**

Surveying shall be conducted at two levels of precision. General site features, such as property boundaries, fence lines, utilities locations, and (petroleum oil and lubricant (POL) pipeline locations shall be surveyed at sub-meter accuracy and precision in Northing, Easting and Elevation. These measurements shall be collected using Global Positioning System (GPS) surveying equipment operated by the Contractor staff. Soil boring locations shall be surveyed using GPS, as well.

Features that require precise elevation controls, such as monitoring wells, stream or ditch bottoms, or reference points, shall be surveyed by a licensed surveyor in the state. Monitoring wells and reference points for stream gauging shall be measured at ground level and at the reference point. Monitoring well reference points shall be at the notch located at this northern fencing point on the PVC casing stickup. Stream gauges shall include a semi-permanent measuring point, depth with top above ground surface. The horizon and the vertical precision for these survey data shall be  $\pm 1$  foot and  $\pm 0.01$  foot, respectively.

Site features and sampling locations shall be surveyed and the results shall be presented in a table containing the name of the feature/Locid, date, reference datum, survey method, Northing, Easting, Elevation, coordinate system, and measurement units. The final survey data shall be submitted in Universal Transverse Mercator (UTM) Projection Zone 17, WGS 84 Meters, NAD 83.

All surveying locations measured by a certified land surveyor in the previously described coordinate system shall be third order (Urquhart, 1962). An XY-coordinate system shall be used to identify locations.

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## **SECTION 2 ENVIROMENTAL SAMPLING**

In general, all sampling activities should include a pre-mobilization phase to ensure sampling equipment and supplies are available onsite during the sampling activity; the subcontracted laboratory project manager is prepared for samples to arrive and be analyzed within site-specific holding times, and data required for specialized data deliverables are available to be input into the Laboratory Information Management System (LIMS). The contract laboratory will provide certified-clean sampling containers properly preserved, shipping containers, packing supplies, de-ionized water for field blanks, chain of custody forms and custody seals. All sampling activities shall include the Site-specific and Base-wide WORK PLANS and the site-specific HASP as reference documents.

Prior to sample collection, Sample Field Forms should be completed to the extent possible, and finalized at each sampling location to track associated quality assurance/quality control (QA/QC) samples, sample times and dates, sample analytical method requirements, and samplers. Each sampling location shall be screened for potential exposure based on the expected target compounds, with general use of the project-specific organic vapor analyzer readings at all sampling locations to be recorded on sampling forms and/or in the field log book. Each sample activity shall require a field log book to be completed, a camera and pictures to be taken to document the activities (noted in the log book), decontamination supplies, containers for IDW, sufficient supplies of new clean, non-powdered disposable nitrile gloves or similar, and other personal protective equipment required in the site-specific HASP. To the extent possible, sampling activities should be conducted with disposable or dedicated sampling equipment to limit the potential for cross-contamination and the need for rinsate blanks.

In addition, material of the sampling devices (e.g., plastic, PVC, metal) discussed below shall be appropriate for the contaminant of concern and shall not interfere with the chemical analyses being performed. These will be described in the site-specific WORK PLAN addenda. All purging and sampling equipment that is not certified clean and disposable shall be decontaminated according to the specifications in Section 1.16 prior to any sampling activities and shall be protected from contamination until ready for use.

### **2.1 DIRECT PUSH DRILLING AND SAMPLING SOP (AI – W ES-01)**

A direct push sampling device is used to collect soil, soil-gas and groundwater samples at specific depths bgs. Direct push systems are hydraulically powered and mounted in a customized four-wheel or track driven vehicle. The base of the sampling device is positioned on the ground over the sampling location and the vehicle is hydraulically raised on the base. As the weight of the vehicle is transferred to the probe, the probe is pushed into the ground. A built-in hammer mechanism allows the probe to be driven through dense materials. Maximum depth penetration under favorable circumstances is about 50 feet.

Soil samples are collected with a specially-designed sample tube. The sample tube is pushed and/or vibrated to a specified depth (approximately one foot above the intended sample interval). The interior plug of the sample tube is removed by inserting small-diameter threaded rods. The sample tube is then driven an additional foot to collect the samples. The probe sections and sample tube are then withdrawn and the sample is extruded from the tube into sample jars.

Soil gas can be collected in two ways. One method involves withdrawing a sample directly from the probe rods, after evacuating a sufficient volume of air from the probe rods. The other method involves collecting a sample through tubing attached by an adaptor to the bottom probe section. Correctly used, the latter method provides more reliable results.

Slotted lengths of probe can be used to collect groundwater samples if the probe rods can be driven to

the water table. Groundwater samples are collected using either a peristaltic pump or a small bailer.

### **2.1.1 Preparation**

Determine extent of the sampling effort, sample matrices to be collected, and types and amounts of equipment and supplies required to complete the sampling effort. Obtain and organize necessary sampling and monitoring equipment. Decontaminate or pre-clean equipment, and ensure that it is in working order. Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan (HASP). Use stakes or flagging to identify and mark all sampling locations. All sample locations should be cleared for utilities prior to commencing intrusive activities.

## **2.2 METHOD 5035 REQUIREMENTS FOR VOCs IN SOIL AND SEDIMENTS SOP (AI-W ES-02)**

Soil and sediment VOC aliquots shall be collected in accordance with Method 5035. Commercially available kits should be used, (such as Terra Core) to account for both low (less than 200 µg/kg) and high level (greater than 200 µg/kg) media. Sample kits should contain the following.

- One disposable calibrated core sampler (5 g);
- One 2-ounce VOA jars with stir bar, 30 mls of Sodium Bisulfate solution and tare weigh for low level analyses; and
- One 2-ounce VOA jars with stir bar, 30 mls of Methanol and tare weight for high level analyses.

The disposable calibrated core sampler shall be used to collect six 5-g plugs from each 3 ft soil core for low level analysis and six 5-g plugs for high-level analysis.

- Plugs for each aliquot should be collected at equi-distance from each other.
  - If a complete 3-foot core is recovered, plugs should be approximately 6-inches apart.
  - If only 2/3 (2 ft) is recovered, plugs should be 4 inches apart.
  - Six plugs are extruded into the low-level analysis container and the container is capped with a Teflon sealed cap (total of 30 g); and
  - Six plugs are extruded into the high-level analysis container and the container is capped with a Teflon sealed cap (total of 30 g).

Jars are immediately sealed with the caps and placed in a plastic bag. No labels or seals are placed on the individual containers as they are pre-weighed and doing so will change the container weight. Labels and seals are instead placed on the plastic bag which holds the VOAs. In addition, the 2-ounce container is filled with the remaining sample to be used to determine the moisture content of the sample so that the result can be provided in dry-weight.

### **2.2.1 ISM Composite Soil Sampling SOP (AI – W ES-05)**

#### **Surface Soil Sampling SOP**

Collection of ISM samples from surface soil and debris areas, and sediment will be accomplished with decontaminated tools such as spades, manual probes, shovels, trowels, and scoops. Surface material will be removed to the required depth and a stainless steel or plastic scoop will be used to collect the samples. Soil and sediment sampling equipment will include:

- Decontaminated stainless-steel sampling spoons or disposable spoons;
- Buckets lined with disposable plastic bags;



- Disposable vinyl gloves;
- Paper towels, wet wipes;
- Decontamination equipment (discussed below); and
- Sample jars, labels, sampling cooler.

The following procedure will be used to collect composite surface soil samples.

1. If present, carefully remove the top layer of vegetation or organic matter to the desired sampling depth interval with a pre-cleaned spade.
2. Obtain the sample from the surface to depth specified in the SAP (e.g., 0 to 6-inch depth), and place into a plastic-lined bucket.
3. A minimum of 30 equally-spaced increments will be required for the ISM composite samples. The increments should be marked using pin-flags or other appropriate markers and outlined to delineate the specific sampling unit or decision unit as applicable, and photo-documented.
4. Particles greater than about 1 cm will be excluded from the initial collection procedure. Gravel, rock, and vegetation will be removed as practical from composited samples as part of the homogenization process.
5. Each composite sample will consist of approximately 1 kg of soil that will be sent to the laboratory in bulk one-gallon bags.
6. Sampling equipment will be decontaminated per Section 3.3 below.

During field sampling activities, AI will record surface conditions that may affect chemical analyses on a Soil Field Sampling Report (Appendix A). These conditions may include:

- Approximate distance to roadways and access roads;
- Observations of seepage;
- Adjacent water level in ditch/stream;
- Distance of sample above surface water surface;
- Soil type (clay, silt, sand, gravel);
- Site features, such as nearby drainage outfalls;
- Obvious deposition of contaminated or clean soil at the site;
- Evidence of dumping or spillage of contaminants; and
  - Soil discoloration, unusual condition of growing plants, and stressed vegetation.

### **2.3 FIELD HEADSPACE SCREENING SOP (AI – W ES-08)**

If initial screening results indicate the presence of organic vapors, a headspace analysis shall be conducted on remaining portions of the sample.

Following collection of split-spoon soil samples, headspace screening shall be performed in the field using a portable PID on the remaining portions of samples selected on the basis of initial screening. Soil samples collected from the borings shall be field screened by filling a self-closing polyethylene plastic bag with approximately 250 grams of soil. The soil samples shall then be vigorously shaken for approximately 30 seconds and allowed to equilibrate a minimum of 15 minutes. The bag headspace shall then be screened for organic vapors by puncturing the bag exterior with the PID probe, inserting the tip to a distance approximately one-half the headspace depth, and recording the highest reading displayed on the instrument meter. The results of field headspace screening shall be recorded on the Drill Log (Appendix A) and used to select samples from each boring for laboratory analysis of selected analytical constituents. The field headspace sample shall be saved for future reference and stored on site.

All information regarding field headspace screening results, soil texture, density, consistency, and color shall be recorded on the Drill Log (Appendix A).

## **2.4 GEOTECHNICAL SAMPLING SOP (AI – W ES – 09)**

During HSA drilling in the relatively soft, moist soil that lies above the saturated zone, geotechnical samples shall be collected, if necessary, in 1 foot long by 3 inches in diameter Shelby tube samplers. Once the FTL has determined that the sampling depth has been reached, the driller shall remove all equipment from the HSA internal casing. The Shelby tube sampler shall then be connected to the end of the drilling rod and lowered into the bottom of the boring, with successive lengths of rod. The full length of the sample shall be pressed into the undisturbed material at the base of the borehole using the hydraulics associated with the Kelly bar. The sampling rod shall then be removed from the borehole and the filled sampler shall be removed from the rod by the drill crew and handed to the sampling technician. The sampling technician shall take a small portion of the material and place it in an evacuated Ziploc™ bag for lithologic logging and headspace assessment. The sampling technician shall cut the Shelby tube sampler to remove unfilled portions of tube, then cover both ends of the sampler with a Teflon™ swatch and plastic cap, label the sampler with information pertaining to the location where the sample was collected, mark the distance bgs associated with each end on the pertinent plastic cap, and store it at the sample station until all geotechnical samples have been collected. The FTL shall then review sampling locations, lithology, and sample integrity and determine which samples shall be sent to the geotechnical laboratory for analysis. Samples shall be stored vertically in the same orientation withdrawn from the borehole. Each sample shall be packed as tightly as possible to minimize impact to the sample integrity. The samples shall be analyzed for geotechnical parameters in accordance with the site-specific WORK PLAN following the appropriate ASTM methods. Geotechnical samples shall be sent to a geotechnical laboratory that has been approved by Facilities and SCDHEC.

## **2.5 SEDIMENT SAMPLING SOP (AI – W ES – 10)**

A potentially more reliable indicator of surface water contamination is the underlying stream sediments. Unlike surface water contamination, which may become diluted or chemically transformed downstream, contaminants have a tendency to accumulate in sediments. Stream sediment samples shall be obtained following collection of surface water samples. The order of stream sediment sampling shall begin with the farthest downgradient sample and move progressively upgradient to minimize potential cross-contamination between locations and media. Where appropriate, stream sediment samples shall be collected from the active streambed on the stream side nearest the contamination source. During surface water and stream sediment sampling, the Contractor field personnel shall sketch the approximate sampling locations and record the following field observations on the Surface Water/Sediment Field Sampling Report (Appendix A):

- Sediment type (e.g., sand, silt, gravel, clay, organic, other);
- The percent recovery, if sediment cores are collected;
- Geomorphology (channel shape, stream bank description, erosional/depositional characteristics);
- Vegetation type;
- Color and/or discoloration;
- Sample depth;
- Odor; and
- The sampling device.

Stream sediment samples shall be collected using a disposable scoop, with VOCs collected directly from the scoop according to Section 2.4 for Method 5035. The remaining non-VOC aliquot will be transferred to a clean disposable homogenization container where excess water will be decanted and the remaining material will be thoroughly homogenized, then transferred to the proper container, sealed, labeled and placed in a cooler on ice to be chilled to 4°C until shipment.

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved as needed, or all samples must be collected unpreserved. To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with deionized water. An appropriate amount of deionized water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured and recorded to allow for the change in the tare weight of each vial (SESDPROC-200-R2, 2010).

## **2.6 SAMPLE HANDLING SOP (AI – W ES – 11)**

### **2.6.1 Sample Containers**

Sample containers are purchased pre-cleaned and treated according to EPA specifications for the methods. Sampling containers shall not be re-used. Containers shall be stored in clean areas to prevent exposure to fuels, solvents, and other contaminants. Amber glass bottles are used routinely where glass containers are specified in the sampling protocol.

### **2.6.2 Sample Volumes, Container Types, and Preservation Requirements**

Sample volumes, container types, and preservation requirements for the analytical methods performed on samples are listed in the Project SAP.

Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times for methods required routinely for work are specified in the SAP.

### **2.6.3 Sample Identification**

A formal sample identification system shall be used to designate each field investigation. The identification system shall provide a tracking procedure to allow retrieval of information about a particular sample location and to ensure that each sample is assigned a unique identification that describes where it was collected.

The sample location identifier consists of up to 10 characters as detailed in the SAP.

### **2.6.4 Sample Custody**

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in field and laboratory records.

The contractor shall maintain COC records for all field and field quality control (QC) samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up or, (4) it is in a designated secure area.

All sample containers shall be sealed in a manner that shall prevent or detect tampering if it occurs. In no case shall tape be used to seal sample containers. The following minimum information concerning the sample shall be documented on the COC form.

- Unique sample identification;
- Date and time of sample collection;
- Source of sample (including name, location, and sample type);
- Designation of matrix spike/matrix spike duplicate (MS/MSD);
- Preservative used;
- Analyses required;
- Name of collector(s);
- Pertinent field data (pH, temperature, etc.);
- Serial numbers of custody seals and transportation cases (if used);
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number (if applicable).



All samples shall be uniquely identified, labeled, and documented in the field at the time of collection as discussed in Section 2.11.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples shall be packed in ice or chemical refrigerant to keep them cool during collection and transportation. During transit, it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. A temperature blank (a 40 ml vial filled with water or similar) shall be included in every cooler and used to determine the internal temperature of the cooler upon receipt of the cooler at the laboratory.

When transferring samples, the individuals relinquishing and receiving shall sign, date, and note the time on the COC record. The analytical laboratory shall maintain a file copy, and the completed original shall be returned to the Contractor PM as part of the final analytical report. This record shall serve to document sample custody transfer from the sampler to the laboratory.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible via overnight courier. Air bills shall be retained as part of the permanent documentation and all sample shipments shall be regulated by the DOT, as described in 49 CFR Parts 171 to 177.

Once the samples have been received by the laboratory, a file of the original documents (e.g., COC forms, special analytical services request form, etc.) pertinent to sample custody and sample analytical protocol shall be maintained, and a copy of the COC file will be sent to the prime Contractor Project Manager.

## **2.7 FIELD QUALITY CONTROL SAMPLES**

### **2.7.1 Equipment Blank**

An Equipment Blank is a sample of distilled water of known quality poured into or over or pumped through a previously used and decontaminated sampling device, collected in a sample container, and transported to the laboratory for analysis. Equipment Blanks shall not be used if sampling equipment clean and dedicated or disposable for single use. Equipment Blanks are used to assess the effectiveness of equipment decontamination procedures. The frequency of collection for Equipment Blanks is specified in the site-specific Project SAP. Equipment Blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

- Equipment blanks shall be designated by “*Site identifier* -EB” and the following characters shall designate which DU and sub DU the equipment blank was collected from.

### **2.7.2 Trip Blank**

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC water samples are taken and are analyzed only for VOC analytes. Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs. Trip blanks shall only be used for non-frozen samples.

- Trip blanks shall be designated by Site Identifier-TB”, and by cooler number and date.
  - Example. TB-01-04122017 is the TB which was in cooler “1” sent on December 4, 2017.



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## **SECTION 3 FIELD MEASUREMENTS**

### **3.1 GENERAL FIELD MEASUREMENT PARAMETERS SOP (AI – W FM – 01)**

The following parameters shall be measured in the field:

- Organic and explosive vapors, using a Photovac 2020 PID or equivalent;
- Water level, using a cable electronic water level indicator or equivalent;
- Site features and certain sampling locations (Soil Gas Survey locations, boreholes, surface soil samples, sediment samples, surface water samples), elevation, and coordinates shall be surveyed using GPS.

### **3.2 FIELD EQUIPMENT CALIBRATION AND QUALITY CONTROL SOP (AI – W FM-02)**

#### **3.2.1 Calibration Frequencies**

All field instruments shall be calibrated on a daily basis, if they are used that day. This does not apply to the PID, which shall be calibrated at least twice per day. In some instances, calibration shall be performed more frequently. Calibration shall provide quality assurance (QA) checks on all field equipment used during implementation of the field investigations. Each instrument shall have an individual identification number. This number shall be transcribed on field data records when using a particular instrument for a sampling event. All calibration, repair, and service records shall be kept in individual equipment log books maintained for each type of instrument. Field equipment that consistently fails to meet calibration standards or exceeds manufacturer's critical limits shall be promptly repaired or replaced. The Contractor shall record equipment calibration on a Field Instrument Calibration Check Form (Appendix A).

#### **3.2.2 Calibration Procedures**

The following are examples of calibration procedures that may be performed during field investigations.

##### **3.2.2.1 Photo Ionization Detector**

The PID shall be calibrated each day prior to the start of field activities. If the PID is in continuous operation, it shall be calibrated at 4-hour intervals. Instrument calibration shall be performed using isobutylene calibration gas of known concentration (100 or 250 ppm). All adjustments to instrument settings shall be recorded in a field log book.

##### **3.2.2.4 Field Quality Assurance / Quality Control Program**

To ensure that sampling and monitoring activities shall meet data quality objectives (DQOs), QC checks shall be implemented for parameters measured in the field. All QC control check information shall be recorded in project-specific field log books and/or forms. The following sections discuss control parameters, control units, and corrective actions for the RI field investigation.

##### **3.2.2.5 Control Parameters**

Several parameters shall be controlled during the field sampling and measurement activities. As previously described, calibration of field instruments and operational checks shall be conducted periodically. The frequency of field control check duplicates shall be a minimum of 10 percent of all field measurements. Temperature, pH, EC, DO, and turbidity shall be checked at the same frequency. As applicable, the materials used to verify control parameter measurements shall be from certified sources. Instrument use, maintenance, and calibration shall follow manufacturer guidelines.

##### **3.2.2.6 Control Limits**



Field Equipment control limits are specified the Project SAP. Field instrument calibration accuracy and duplicate precision for field measurements must meet acceptance criteria, or instrument readings shall be considered suspect. Appropriate corrective actions shall be taken whenever field instruments fail to meet acceptance for accuracy and precision.

#### 3.2.2.7 Corrective Action

Corrective action for all field instruments shall involve a review of the operator's manual. If necessary, instrument maintenance and repairs shall be performed as corrective actions in addition to normally scheduled maintenance operations. Any maintenance shall be recorded on a Field Instrument Calibration Check Form (Appendix A).

### 3.3 EQUIPMENT MAINTENANCE AND DECONTAMINATION SOP (AI – W FM-03)

All field measurement equipment shall be decontaminated according to the specifications in Section 1.16 prior to any measurement activities and shall be protected from contamination until ready for use.

### 3.4 ORGANIC AND EXPLOSIVE VAPOR MEASUREMENT SOP (AI – W FM – 04)

During borehole advancement, the air in the breathing zone of on-site personnel shall be evaluated for the presence of organic (e.g., VOCs, SVOCs, petroleum hydrocarbons) using a Photovac 2020 PID or, or equivalents. Air monitoring data shall be tabulated on a Health and Safety Exposure Monitoring Form from the site-specific HASP. Procedures provided in the site-specific HASP shall be followed. In addition to monitoring the breathing zone around the borehole, the PID shall be used to screen for organic vapors when performing field headspace screening of split-spoon soil samples.

### 3.5 GROUND – WATER LEVEL MEASUREMENT SOP (AI – W FM-05)

Water-level measurements shall be taken in all wells and piezometers to determine the elevation of the water table or piezometric surface at least once within a single 24-hour period. These measurements shall be taken after all wells and piezometers have been installed and developed and their water levels have recovered completely. Any conditions (e.g., barometric pressure) that may affect water levels shall be recorded in the field log. The field log shall also include the previous water level measurement for each well (to determine if current water level is reasonable).

Water-level measurements shall be taken with electric sounders, air lines, pressure transducers, or water-level recorders (e.g., Stevens's recorder). Devices that may alter sample composition shall not be used. Pressure gauges, manometers, or equivalent devices shall be used for flowing wells to measure the elevation of the piezometric surface. All measuring equipment shall be decontaminated according to the specifications in Section 1.16. Groundwater level shall be measured to the nearest 0.01 foot. (Two or more sequential measurements shall be taken at each location until two measurements agree to within + or -0.01 foot.)

Static water levels shall be measured each time a well is sampled, and before any equipment enters the well. If the casing cap is airtight, allow time prior to measurement for equilibration of pressures after the cap is removed. Repeat measurements until water level is stabilized.

Water level measurements shall be taken from existing and newly installed groundwater monitoring wells prior to well development and sample purging. Groundwater levels in the bedrock groundwater monitoring wells shall be recorded to the nearest 0.01 foot using a cable electronic water level indicator or the equivalent. The primary objectives of measuring water levels in the groundwater monitoring wells shall be to estimate the volume of water to facilitate well development and sample purging, and to provide a preliminary evaluation of principal groundwater flow directions. The total depth of each groundwater monitoring well shall also be measured after installation to evaluate its usefulness for future

monitoring.

## **SECTION 4 RECORD KEEPING**

### **4.1 GENERAL RECORD KEEPING SOP (AI-W RK-01)**

AI shall maintain field records sufficient to recreate all sampling and measurement activities and to meet all Project data loading requirements. The requirements listed in this section apply to all measuring and sampling activities. Requirements specific to individual activities are listed in the section that addresses each activity. These records shall be archived in an easily accessible form and made available to the Facility RPM upon request.

The following information shall be recorded with indelible ink in a permanently bound notebook with sequentially numbered pages for all field activities:

- Location,
- Date and time,
- Identity of people performing activity, and
- Weather conditions.

For field measurements, the numerical value and units of each measurement, and the identity of and calibration results for each field instrument shall also be recorded on field logs.

The following additional information shall be recorded for all sampling activities:

- Sample type and sampling method;
- The identity of each sample and depth(s), where applicable, from which it was collected;
- The amount of each sample;
- Sample description (e.g., color, odor, clarity);
- Identification of sampling devices; and
- Identification of conditions that might affect the representativeness of a sample (e.g., refueling operations, damaged casing).

Records shall be kept for all activities associated with the field activities, as a means to maintain full documentation of project QA/QC procedures and compliance. In general, all documents shall be completed in permanent black ink. Errors shall be corrected by crossing them out with a single line and then dating and initialing. The use of correction fluids shall not be allowed. The documents used during the field investigation shall remain on site (if possible) during the entire effort so that they can be reviewed by interested parties. Forms shall be kept organized and in a central file also located on site, if applicable. Records shall be kept in the form of field log books and standardized forms which have been included as Appendix A.

### **4.2 Log Book SOP (AI – W RK – 02)**

The Contractor field personnel shall maintain a field log book during the investigation. The field log book is the master field investigation document that is a bound book with a hard cover and sequentially numbered pages. The primary objective of the field log book is to maintain, within one document, the actual field data or references to other field documents that contain a specific description of every activity that has occurred in the field on any given day. Any administrative occurrences, conditions, or activities that have affected the field work shall be recorded in the field log book.

All field activities entered into the field log book shall be signed and dated by the responsible party. The following is a list of the type of information that shall be recorded in the field log book:

- Name and title of author, date and time of entry, and physical/environmental conditions during the field activity;
- Name and address of field contact;
- Name and titles of field crew;
- Name and titles of all site visitors;
- Documentation of Health and Safety activities;
- Type of sampled media (e.g., soil, groundwater);
- Number and volume of samples taken;
- Description of sampling points;
- Date and time of overall sample collection;
- Sample identification numbers;
- References, maps and descriptions of photographs;
- General decontamination procedures;
- Instrument calibration;
- Records of telephone conversations; and
- Weather conditions

#### **4.3 FIELD DATA FORMS SOP (AI – W RK – 03)**

In addition to the above-referenced field log book, the Contractor shall complete and maintain standardized field data forms for specified field activities. Field data forms are included in Appendix A. They consist of the following:

- Employee/Visitor Daily Roster;
- Tailgate Safety Meeting Report;
- Field Instrument Calibration Check Form;
- Field Sampling Report (Soil/Sediment);
- HTRW Drill Log (Eng. Form 5056-R);
- Borehole Abandonment Log;
- Waste Inventory Tracking Log
- Quality Assurance Report Daily Log (Eng. Form 2538-1);

## SECTION 5 REFERENCES

- American Society for Testing and Materials (ASTM). 1999. ASTM D-1586. Standard Test Method for Penetration Test and Split-Spoon-Barrel Sampling of Soils. (D-1586-99).
- ASTM. 1990. ASTM D-2488-90. Standard Practice for Description and Identification of Soils (Visual-Manual Procedure).
- Tennissen, A.C. 1983, Nature of Earth Materials, 2nd Edition, p. 204-348.
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- Environmental Protection Agency (USEPA), 1993. Suggested Operating Procedures for Aquifer Pump Tests. EPA/540/S-93/503. February
- USEPA, 1994. Slug Test SOP # 2046. November.
- USEPA, 1996. Soil Gas Sampling SOP # 2042. June.
- USEPA Region 4, 2007. Groundwater Sampling Operating Procedure, Science and Ecosystems Support Division (SESD) SESDPROC-301-R1. November.
- USEPA Region 4, 2007. Surface Water Sampling Operating Procedure, Science and Ecosystems Support Division (SESD) SESDPROC-201-R1. November.
- USEPA Region 4, 2010. Sediment Sampling Operating Procedure, SESDPROC-200-R2. September





## Appendix D – Laboratory Standard Operating Procedures



## Document Information

<b>Document Number:</b> ENV-SOP-MTJL-0112	<b>Revision:</b> 04
<b>Document Title:</b> Multi-Increment Sampling	
<b>Department(s):</b> SVOA	

## Date Information

<b>Effective Date:</b> 08 Sep 2020
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## Notes

<b>Document Notes:</b>
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All dates and times are in Central Time Zone.

**ENV-SOP-MTJL-0112****QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Michael Jones (006596)	Quality Analyst 1	08 Sep 2020, 07:46:49 AM	Approved

**Management Approval**

Name/Signature	Title	Date	Meaning/Reason
James Burns (006456)	Manager - EHS	02 Sep 2020, 10:37:09 AM	Approved
Christopher Johnson (006487)	Manager - Operations	03 Sep 2020, 12:13:06 PM	Approved



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**TEST METHOD STANDARD OPERATING PROCEDURE**

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**TITLE:** Multi-Increment Sampling**TEST METHOD:** NA**ISSUER:** Pace Analytical National Center for Testing & Innovation

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## **1.0 Scope and Application**

- 1.1 Appendix A of EPA Method 8330B (SW-846) specifically addresses field sampling. The appendix provides guidance for explosive residue sample collection, handling, and laboratory processing techniques. Method 8330B recommends the use of multi-increment (MI) sampling, which involves the extraction of a representative portion of material from within a single decision unit which will adequately address potential compositional and distributional heterogeneity. In MI sampling, several increments from the same decision unit are combined to form one sample that is submitted for laboratory analysis. The procedures for MI sampling are specifically designed to minimize sampling error and provide a more scientifically-representative mean concentration of the contaminant(s) present in the decision unit.
- 1.2 Initial demonstration for achieving samples size below 75µm per DOD/DOE QSM is on file in the QA department.

## **2.0 Summary of Method**

- 2.1 Samples are dried, ground, and homogenized before subsamples are taken for sample preparation.

## **3.0 Interferences**

- 3.1 Care must be taken to not cross-contaminate samples during the drying, sieving, and grinding procedures. Grinding blanks are required to verify procedure is free from cross contamination.
- 3.2 The drying process may result in quantitative losses of some analytes. Project Managers may consider eliminating the drying process prior to analysis or removing poor performers from the target analyte list if drying is required.

## **4.0 Definitions**

- 4.1 Sieve: A device made of wire mesh held in a frame through which finer particles of a mixture of various sizes may be passed to separate them from coarser ones or through which soft materials may be forced for reduction to fine particles.
- 4.2 Shatterbox: A device for mechanically pulverizing a sample or material.
- 4.3 Ball Mill: A device using ceramic pellets and rotation in a closed container to pulverize the contents.
- 4.4 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

## **5.0 Health and Safety**

- 5.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.






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- 5.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.
- 5.3 Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- 5.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handling these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.
- 5.5 Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

## 6.0 Sample Collection, Preservation, Holding Time, and Storage

- 6.1 Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.
- 6.2 Pace National will typically receive samples in 4-8oz containers for processing.

## 7.0 Equipment and Supplies

- 7.1 Sieve: 10mesh
- 7.2 Grinder: Shatterbox or equivalent capable of reducing particle size to <75µm
- 7.3 Drying rack

## 8.0 Reagents and Standards

- 8.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted.
- 8.2 A weekly check sample is purchased and ran through the entire process, except drying, as allowed per method. Acceptance criteria is determined by the company supplying the standard and will change with each lot received.

## 9.0 Procedure

- 9.1 All sample contents within the container are emptied in to a pan/weigh boat and dried to a constant weight.

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**TEST METHOD STANDARD OPERATING PROCEDURE**


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**TITLE:** Multi-Increment Sampling

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- 9.1.1 A Blank matrix must be dried with samples.
  - 9.1.2 Obtain a clean pan/weigh boat and record the tare weight.
  - 9.1.3 Empty the entire contents of the sample container into the pan/weigh boat.
  - 9.1.4 Using gloved hands break the soil into small pieces as necessary to facilitate the drying process. Use fresh gloves for each sample to prevent cross contamination.
  - 9.1.5 Record the initial weight of the entire sample.
  - 9.1.6 After the initial weight is obtained, dry the sample at room temperature in a hood for approximately 24 hours. Then obtain a 2nd sample weight.
  - 9.1.7 Continue the drying process for approximately 12 hours and obtain a 3rd sample weight.
  - 9.1.8 Two consecutive weights of less than 10% difference, taken approximately 12 hours apart, is considered to be dried to a constant weight.
  - 9.1.9 Dates/Times are recorded as well as the ambient temperature with each weighing of samples.
  - 9.2 For all methods or when client-specific data quality objectives (DQOs) require grinding, dried sample is introduced into the shatterbox or equivalent. The entire sample must be ground. If multiple portions are ground separately, the aliquots must be combined prior to subsampling for extraction. Samples are ground up to Three minute intervals. Intervals and duration are dependent on the sample matrix and analytes of interest for the specific project. The Blank and weekly check sample must also proceed through this step.
  - 9.3 Dried sample material is passed through a 10mesh (2mm) sieve (may be assisted using gloved hands). Do not intentionally include vegetation unless project specifications include this requirement. Depending on sample matrix, sieving may be performed initially to facilitate the drying process.
  - 9.4 The Blank matrix is ground at the end of each batch. A blank will also be ground after any sample of known concentration above detectable limits, including quality control samples.
  - 9.5 Each sample/QC is spread into a pan in order to perform sufficient subsampling of the final sample aliquot. At least 30 sample increments must be taken for the subsampling procedure. The sample volume extracted for analysis should represent the entire ground sample.
- NOTE: If sample volume does not allow 30 aliquots, a note will be made on the extraction log.
- 9.6 Add surrogate to each sample/QC and spike MS/D after the initial extraction weight is acquired.
  - 9.7 See the specific method extraction SOP for further processing information.

## 10.0 Data Analysis and Calculations

- 10.1 See the Laboratory Quality Assurance Manual for equations for common calculations.

## 11.0 Quality Control and Method Performance

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**TEST METHOD STANDARD OPERATING PROCEDURE**


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### 11.1 Analyst Qualifications and Training

- 11.1.1 Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications for Chemistry* for more information.

## 12.0 Data Review And Corrective Action

### 12.1 Data Review

- 12.1.1 Pace National's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.
- 12.1.2 The review steps and checks that occur as employees complete tasks and review their own work is called primary review.
- 12.1.3 All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace National's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.
- 12.1.4 A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.
- 12.1.5 Refer to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

### 12.2 Corrective Action

- 12.2.1 Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.




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**TEST METHOD STANDARD OPERATING PROCEDURE**
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### 13.0 Pollution Prevention and Waste Management

13.1 Pace National proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

13.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace National's Chemical Hygiene Plan / Safety Manual.

### 14.0 Modifications

14.1 Pace National is set up currently to process from 4oz/8oz/16oz/32oz jars that have been prepared in the field from bulk containers. Pace National cannot currently process bulk samples for this method.

14.2 Due to limited sample volume received as listed in 14.1:

14.2.1 Duplicate subsampling is performed rather than triplicate

14.3 A weekly check sample is performed to verify the MIS process in lieu of processed LCS.

### 15.0 Responsibilities

15.1 Pace National employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace National's policy for temporary departure.

15.2 Pace National supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

### 16.0 Attachments

16.1 Not applicable to this SOP

### 17.0 References

17.1 Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC), SW-846 Method 8330B, Revision 2, October 2006, Appendix A.

17.2 Quality Systems Manual (QSM) for Environmental Laboratories, Department of Defense (DoD), Version 5.1, 2017.






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## 18.0 Revision History

### Current Version (Pace National):

Date	Description of Revisions
9/1/2020	Technical and quality review. Deleted sections 9.2 and 9.3. Added sections 9.2 and 9.3. Revised section 14.1.

### Superseded Versions (ESC Lab Sciences SOP #330377):

Version	Date	Description of Revisions
1	4/1/2016	SOP Origination.
2	4/17/2017	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 2.1.2, 3.1, 8.1, 8.3, 9.0, 13.0, 14.1, and 14.2.
3	7/31/18	Technical and quality review and update. Changed logo. Revised Sections 8.3, 8.4, 8.5, 11.2. Added Sections 7.1, 8.1.1-8.1.9, 13.2, and 13.3

### Superseded Versions (Pace National):

Date	Description of Revisions
7/31/2019	Converted SOP over to new format. Revised section 9.5. Added section 1.2. Deleted sections 9.4, 14.2.2 and renumbered as necessary.
7/16/2020	Technical and quality review.

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## Document Information

<b>Document Number:</b> ENV-SOP-MTJL-0215	<b>Revision:</b> 08
<b>Document Title:</b> Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)	
<b>Department(s):</b> Metals	

## Date Information

<b>Effective Date:</b> 28 Aug 2020
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## Notes

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**Signature Manifest****Document Number:** ENV-SOP-MTJL-0215**Revision:** 08**Title:** Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

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**ENV-SOP-MTJL-0215****QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Regina Stone (006490)	Quality Analyst 3	26 Aug 2020, 09:32:37 AM	Approved

**Management Approval**

Name/Signature	Title	Date	Meaning/Reason
Jeremiah Gupton (006426)	Manager	26 Aug 2020, 10:04:42 AM	Approved
James Burns (006456)	Manager - EHS	26 Aug 2020, 01:21:09 PM	Approved
Heidi Ferrell (006481)	Manager - Operations	26 Aug 2020, 03:36:32 PM	Approved



## STANDARD OPERATING PROCEDURE

**TITLE:** Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

**ISSUER:** Pace National – Mt. Juliet, Tennessee

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### 1.0 SCOPE AND APPLICATION

**STATE NOTE:** For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0214.

- 1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B, 6010C, and 6010D for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other digestates after appropriate preparatory procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C/D from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed in Table 1.2b.

- 1.2 This method is applicable for the analytes listed in Table 1.2a and b. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by Pace Analytical National Center for Testing & Innovation (Pace National).

**Table 1.2a: Environmental Analytes and Reporting Limits** (Subject to change, see section 13.1)

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200 .7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/ D	RL	Units
Aluminum	✓	✓	00.200	mg/L	✓	2.00	mg/Kg
Antimony	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Arsenic	✓	✓	00.010	mg/L	✓	1.00	mg/Kg
Barium	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Beryllium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Boron	✓	✓	0.050	mg/L	✓	5.0	mg/Kg
Cadmium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Calcium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Chromium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Cobalt	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Copper	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Iron	✓	✓	0.100	mg/L	✓	10.0	mg/Kg
Lead	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Lithium	✓		0.015	mg/L	✓	1.50	mg/Kg
Magnesium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Manganese	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Molybdenum	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Nickel	✓	✓	0.010	mg/L	✓	1.00	mg/Kg

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**ISSUER:** Pace National – Mt. Juliet, Tennessee

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Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Potassium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Selenium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Silicon	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Silver	✓	✓	0.005	mg/L	✓	5.00	mg/Kg
Sulfur	✓	✓	1.0	mg/L	✓		mg/Kg
Sodium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Strontium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Thallium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Tin	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Titanium	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Vanadium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Zinc	✓	✓	0.050	mg/L	✓	5.00	mg/Kg

\*May not meet required Drinking Water Maximum Contamination Levels (MCLs) using this methodology.

**Table 1.2b: Hardness Categories and Reporting Limits**

(Subject to change, see section 13.1)

Hardness:	RL (mg/L)
Calcium Hardness	1.25
Magnesium Hardness	0.41
Total Hardness	1.6

- 1.3 For the determination of total recoverable analytes in aqueous and solid samples, an acid digestion process is required. Environmental samples for analysis by Method 6010B, 6010C, or 6010D including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in ENV-SOP-MTJL-0217 and ENV-SOP-MTJL-0219.
- 1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.
- 1.5 To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be <0.2% (w/v).



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- 1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of <1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria<sup>14.2</sup>.

Method 6010D – Samples that are not digested necessitate the use of either an internal standard or should be matrix-matched with the standards. If using the former option, the instrument software should be programmed to correct for the intensity differences of the internal standard between samples and standards. **NOTE:** All samples analyzed by Method 6010 are typically digested.

- 1.7 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon™) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube<sup>14.2</sup>.
- 1.8 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.
- 1.9 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation<sup>14.2</sup>.
- 1.10 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used.

Method 6010D – IDLs are necessarily instrument-specific. Therefore, if needed, an IDL must be determined through a separate experimental study for each instrument. IDLs should be established, at a minimum, on an annual basis for each matrix and for each preparatory/determinative method combination used.

**TABLE 1.10: WAVELENGTHS**  
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Radial
Antimony	206.836	Axial
Arsenic	188.979	Axial
Barium	233.527	Axial
Beryllium	313.107	Radial
Boron	249.772	Radial
Cadmium	214.440	Axial
Calcium	317.933	Radial




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Analyte	Wavelength (nm)	Type of View
	373.690	
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Radial
Iron	259.940 271.441	Radial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Radial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Phosphorus	177.495	Axial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592 818.326	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial
Tin	189.927	Axial
Titanium	334.940	Radial
Vanadium	292.402	Radial
Zinc	213.857	Axial

- 1.11 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.
- 1.12 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Operations Manager and Project Manager is required for each modification and Regulatory Affairs approval must also be secured for any deviation.
- 1.13 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ENV-SOP-MTJL-0016. Updated MDL records are filed and stored in a central location within the department.




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1.13.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD), and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DoD support; then the frequency of these studies must meet the requirements of the current DoD QSM.

1.14 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed semi-annually for each analytical instrument or when there are major changes/repairs to the instrument<sup>14.5, 14.1</sup>. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument<sup>14.5</sup>.

## 2.0 METHOD SUMMARY AND DEFINITIONS

2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

2.2 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.

2.3 Total (Total Recoverable) Analyte – The concentration of analyte determined either by “direct analysis” of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method

2.4 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength. The IDL assures with 99% certainty that a value is above the instrument noise level.






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**Note:** An IDL is a statistical determination without analytes present used to assess background correction protocols and an MDL is determined with low levels of analytes present to determine instrument sensitivity for each analyte.

- 2.5 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration remains linear.
- 2.6 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.
- 2.7 Interference Check Sample (ICS) – A series of five solutions (ICSA, ICSAB, ICSA2, LA, & CE) to verify that inter-element interferences are correctly compensated. The ICS checks provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7
  - 2.7.1 ICSA – A solution containing only the interfering analytes at high concentrations.
  - 2.7.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.
  - 2.7.3 ICSA2 – A solution containing interfering analytes not contained in the ICSA.
  - 2.7.4 LA – A solution containing Lanthinum at a high concentration.
  - 2.7.5 CE – A solution containing Cerium at a high concentration.
- 2.8 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.
- 2.9 Preparation Batch - For method 6010B/C/D/ EPA 200.7 (WW only): A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7 (DW only): A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.
- 2.10 Analytical batch - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.
- 2.11 Inter-element correction (IEC) coefficient - analyte concentration equivalent arising from a given interferent's concentration.
- 2.12 Serial Dilution - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.
- 2.13 Post Spike – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.
- 2.14 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D; the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.




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- 2.15 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

### 3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- 3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.
- 3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.
- 3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handling these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.
- 3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.
- 3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.
- 3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

### 4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the




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client approves the completion of the analytical process, sample results can be qualified per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

- 4.3 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.
- 4.4 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH <2.
- 4.5 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH <2. Preservation may be done at the time of collection; however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at <2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.
- 4.6 Solid samples require no preservation prior to analysis. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.
- 4.7 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is six (6) months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid.

## 5.0 INTERFERENCES

- 5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
  - 5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument




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method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferents as well (ICSAB solution may be used).

- 5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involve measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and are reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.
- 5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at a high concentration within the LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.
- 5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.
- 5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that






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better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching and by standards addition procedures. Chemical interferences are highly dependent on matrix type.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.

5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard for each analyte cannot be greater than the LDR for that analyte. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.

5.6 Background correction is performed as needed and LDR and IEC studies are completed as required by each published analytical method and whenever significant changes to instrumentation are made. Background, or blank matrix, subtraction is not performed for environmental samples.

## 6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively coupled plasma emission spectrometer:

6.1.1 Perkin Elmer Model 5300 or Thermo Model 7000 series ICP, or equivalent, with background correction and computer control

6.1.2 Cetac Autosampler or ESI autosampler

6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.

6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities

6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity

6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2 $\mu$ L to 5000 $\mu$ L

6.5 Class A volumetric flasks for standards preparations

6.6 Polypropylene (PP) conical tubes



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6.7 Peristaltic pump

### 7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected unless otherwise noted.

7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl

7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.

7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO<sub>3</sub>

7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO<sub>3</sub> to 400mL reagent water and dilute to 1L with reagent water.

7.4 Laboratory Reagent water

7.5 ICP Standard List

Calibration	Stock Std Cat #	Working Standard Preparation
RL Standard #1	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
0.5ppm Standard #2	HP6928-1L	125mL of 2.0ppm Std, 10% rinse to volume 500mL
1ppm Standard #3		250mL of 2.0ppm, 10% rinse to volume 500mL
2ppm Standard #4		Direct pour
10ppm Standard #5	HP6409-500	Direct pour
250ppm Standard #6	HP4526-1L	500mL of 500ppm Std, 10% rinse to volume 1000mL
500ppm Standard #7		Direct pour
La Standard #8	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL

Standards	Stock Std Cat #	Working Standard Preparation
ICV	ESC-8	5mL of Stock ICV Std, 10% rinse to volume 500mL
CCV	HP6929-1L	50mL of Stock CCV Std, 10% rinse to volume 1000mL
ICVLL	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
CCVLL		50mL of Stock LL Std, 10% rinse to volume 1000mL



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Standards	Stock Std Cat #	Working Standard Preparation
ICSA	ICL500-6	50mL of Stock A Std, 10% rinse to volume 500mL
ICSAB	ICL500-6,HP2739-500	50mL of Stock A Std, 5ml of Stock AB Std, 10% rinse to volume 500mL
CE	HP100010-1	5mL of Stock Ce Std, 10% rinse to volume 500mL
ICSA2	varies	5 mL of Stock Std, 10% rinse to volume 500mL
LA	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL.
IEC / LDR As	HP10003	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Co	HP100013	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cr	HP100012	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cu	HP100014	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Mn	HP100032	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Si	HP100050	10mL of Stock As Std, 10% rinse to volume 1000mL
Internal Std	HP10M67-1,HP10M24-1	1mL of Stock Y, 3mL of Stock In, 10% rinse to volume 1000mL
10% Blank and Rinse	A200C-212	Fill container to 90% with DI water, add HNO <sub>3</sub> to volume

### 7.6 Blanks - Three types of blanks are required for ICP-AES analysis

7.6.1 The calibration blank is used to establish the baseline for the instrument prior to the analysis of the analytical curve. The calibration blank is prepared by acidifying reagent water to the same acid concentration as used for the standards (10% HNO<sub>3</sub>).

**NOTE:** The calibration blank must be stored in a FEP bottle to minimize leaching from other container materials that can cause an elevation in the target analytes leached causing an inherent bias in the calibration and quantitation of field samples when baselines are established prior to calibration of the ICP-AES.

7.6.1.1 Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to field sample analyses. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.

7.6.2 The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be



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prepared in the same manner as the samples including sample digestion, when applicable.

7.6.3 The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank (10% HNO<sub>3</sub>). This solution is stored in a convenient manner. The rinse blank is used for equipment “wash out” to flush the sample delivery system and eliminate memory effects (carryover) from previous samples or standards.

7.7 Mixed Calibration Standard Solutions are used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. See section 7.5 above for preparation of these standards.

**Concentration of Target Analytes in Calibration Standards in mg/L**

Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Silver	0.005	0.5	1.0	2.0				
Aluminum	0.2				10	250	500	
Arsenic	0.01	0.5	1.0	2.0				
Boron	0.2		1.0	2.0				
Barium	0.005	0.5	1.0	2.0	10			
Beryllium	0.002	0.5	1.0	2.0				
Calcium	1.0		1.0	2.0	10	250	500	
Cadmium	0.002	0.5	1.0	2.0				
Cerium						10		
Cobalt	0.01	0.5	1.0	2.0				
Chromium	0.01	0.5	1.0	2.0				
Copper	0.01	0.5	1.0	2.0				
Iron	0.10	0.5	1.0	2.0	10	100	200	
Potassium	1.0			2.0	10	50	100	
Phosphorus	0.1	0.5	1.0	2.0				
Lanthanum								10
Lithium	0.015	0.5	1.0					
Magnesium	1.0				10	250	500	
Manganese	0.01	0.5	1.0	2.0				
Molybdenum	0.005	0.5	1.0	2.0				
Sodium	1.0		1.0	2.0	10	250	500	
Nickel	0.01	0.5	1.0	2.0				
Lead	0.005	0.5	1.0	2.0				
Antimony	0.01	0.5	1.0	2.0				
Selenium	0.01	0.5	1.0	2.0				
Silicon	0.2	0.5	1.0	2.0	10			
Strontium	0.01	0.5	1.0	2.0				
Sulfur	1.0		1.0	2.0	10	50	100	
Tin	0.05	0.5	1.0	2.0				
Thallium	0.01	0.5	1.0	2.0				
Vanadium	0.02	0.5	1.0	2.0				
Zinc	0.05	0.5	1.0	2.0				
Titanium	0.05	0.5	1.0	2.0				





## STANDARD OPERATING PROCEDURE

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- 7.8 Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level ICV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	1.0
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	1.0
Beryllium	1.0
Calcium	10.0
Cadmium	1.0
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	10.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	1.0
Sodium	10.0
Nickel	1.0
Lead	1.0
Antimony	1.0
Selenium	1.0
Silicon	1.0
Strontium	0.4
Tin	1.0
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	10.0

- 7.9 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level CCV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	0.5
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	0.50
Beryllium	0.20
Calcium	50.0
Cadmium	0.50
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	50.0
Lithium	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	0.25
Sodium	50.0
Nickel	1.0
Lead	0.50
Antimony	0.5
Selenium	1.0
Silicon	2.0
Strontium	1.0
Tin	0.5
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0



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Analyte	Concentration
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Sulfur	5.0

- 7.10 Low Level Initial/Continuing Calibration Verification for EPA 6010C (ICVLL/CCVLL) –The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards. See Section 7.5 above for the preparation of this standard.

The concentration of the low-level ICVLL/CCVLL solution is listed in the table below:

Analyte	Concentration (mg/L)
Silver	0.005
Aluminum	0.2
Arsenic	0.01
Boron	0.2
Barium	0.005
Beryllium	0.002
Calcium	1
Cadmium	0.002
Cobalt	0.01
Chromium	0.01
Copper	0.01
Iron	0.10
Potassium	1
Lithium	0.015
Magnesium	1
Phosphorus	0.1

Analyte	Concentration (mg/L)
Manganese	0.01
Molybdenum	0.005
Sodium	1
Nickel	0.01
Lead	0.005
Antimony	0.01
Selenium	0.01
Silicon	0.2
Strontium	0.01
Tin	0.05
Thallium	0.01
Vanadium	0.02
Zinc	0.05
Titanium	0.05
Sulfur	1.00

- 7.11 Interference Check Solutions (ICSA, ICSAB, ICSA2, LA, CE) – The ICS checks are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA, ICSA2, LA, and CE solutions contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 and 6010D refers to this standard as the Spectral Interference Check (SIC) standard. See Section 7.5 above for the preparation of these standards.

7.11.1 The ICSA solution contains 5000mg/L of each Al, Ca, Mg and 2000mg/L Fe.

7.11.2 The ICSAB solution contains all the components at the same concentrations of the ICSA and other target analytes of interest spiked. In the working ICSAB solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. All other analytes (arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, tin, thallium, vanadium and titanium) are present at 0.5mg/L.

7.11.3 The ICSA2 solution contains interfering elements that are not in the ICSA. In the working ICSA2 solution, all analytes are present at 10mg/L.




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7.11.4 The LA solution contains 10mg/L of La.

7.11.5 The CE solution contains 10mg/L of Ce.

7.12 The IEC / LDR solutions are prepared to contain known concentrations of interfering elements that are used to adjust the IECs and are used for daily LDRs for 6010D. The interfering elements are present at 10mg/L.

7.13 Internal Standards – The internal standard response is used to measure the relative responses of other method analytes in each sample. Yttrium and Indium are used as internal standards. See Section 7.5 above for the preparation of this standard.

7.14 For the preparation of Laboratory Control Samples (LCSs) and Matrix Spikes (MSs) see the applicable sample preparation SOPs.

## 8.0 PROCEDURE

### 8.1 Sample Analysis

8.1.1 **Initializing the Instrument:** Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance.

8.1.1.1 Replace the uptake tubing daily.

8.1.1.2 If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than five (5) minutes. If cleaning is successful, dry the part using compressed air or argon and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.

8.1.2 **Instrument Stability:** The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 30 minutes of operation.

8.1.3 **Instrument Calibration:** For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. A peristaltic pump is used to introduce all solutions, samples, and the internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before obtaining the sample analyte response.

8.1.3.1 Use the average value from three replicate analyte responses per sample to be correlated to the overall analyte concentration in the solution being sampled. Flush the system with the rinse blank for a minimum of 60 seconds between each standard.




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8.1.3.2 The calibration regression is generated using first order linear regression of a calibration blank and three calibration standards where each element is present. The blank is included as a point in the calibration curve to determine the baseline correction needed for the instrument to effectively quantitate target analyte concentrations.

8.1.3.3 Calibration acceptance criteria are described in section 10.4.

8.1.4 **Internal Standard:** All standards/samples/QC etc. contain yttrium and indium as the internal standards. The instrument adds the internal standards automatically. The instrument injects a constant volume into each solution being analyzed (i.e. standard, blank, field sample, LCS/LCSD/MS/MSD/DUP) and monitors the intensity at the sample level. An internal standard is the chosen alternative to the method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by an internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.14.

8.1.5 **Calibration Accuracy:** Verify the acceptable initial calibration of the instrument using a standard source that is either an independent lot or entirely different manufacturer to ensure calibration accuracy. After calibrating and rinsing the instrument, analyze the ICV and, if analyzing samples using EPA 6010C, analyze the ICVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.

8.1.6 **On-going Calibration Stability:** Verify the acceptable on-going instrument calibration by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples using the CCV and CCB and at the end of the analytical run using the CCV, CCVLL (if analyzing EPA 6010C samples), and CCB that must meet the criteria described in sections 10.6 & 10.14.

8.1.7 **Accurate Background Corrections:** The interference check standards (ICSA, ICSAB, ICSA2, LA, and CE) are used to verify the inter-element and background correction factors at the beginning of an analytical run. The ICSA and ICSAB are verified during every 8-hour work shift. The interference check standards must meet the criteria found in section 10.10.

8.1.8 An Initial Calibration Blank (Section 7.5.1) is analyzed before sample analysis is initiated to verify the cleanliness of the analytical system. Acceptance criteria are described in section 10.14.

8.1.9 **Field Sample Analysis:** After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.

8.1.10 **Dilutions:** If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte's determined Linear Dynamic Range (LDR), see section 10.4.2 for further guidance.





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### 9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/kg dry weight corrected for solid samples.
- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentrations.
- 9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentration. Routine reporting limits are adjusted for any dilution required by the sample analysis.
- 9.4 Results are reported to Three significant figures by the laboratory LIMS. Analyte concentrations for solids data should be rounded in a similar manner following dry weight corrections.
- 9.5 For total recoverable analytes in solid samples, calculate the target analyte concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL based on additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg) = dry-weight basis} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)  
 V = Volume of extract (L, 100mL = 0.1L)  
 D = Dilution factor (undiluted = 1)  
 W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

- 9.6 Soil samples are routinely reported on a dry weight basis. Soil samples must be processed using the ENV-SOP-MTJL-0065, *Total Solids*. After a dry weight for each sample has been obtained, the calculations are performed automatically by the laboratory LIMS as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried  
 WW = Sample weight (g) before drying

- 9.7 Hardness calculations:

Total Hardness, mg equivalent  $\text{CaCO}_3/\text{L}$  = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]  
 Calcium Hardness = 2.497 [Ca, mg/L]  
 Magnesium Hardness = 4.118 [Mg, mg/L]



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- 9.8 To calculate the silica concentration from silicon analysis:

$$\text{Silica (mg/L)} = 2.14 * [\text{Silicon, mg/L}]$$

- 9.9 Formula needed to calculate dilution of stock standards of known concentration to a known final volume, using the basic chemistry formula,  $C_1 * V_1 = C_2 * V_2$ :

$$V_{\text{stock}} = V_{\text{std}} * C_{\text{std}} / C_{\text{stock}}$$

where:  $V_{\text{stock}}$  = volume of stock standard required (mL)

$V_{\text{std}}$  = final volume of diluted standard required (mL)

$C_{\text{stock}}$  = concentration of stock standard required ( $\mu\text{g/mL}$  or  $\mu\text{g/L}$ )

$C_{\text{std}}$  = final concentration of diluted standard required ( $\mu\text{g/mL}$  or  $\mu\text{g/L}$ )

**NOTE:** Be sure to maintain consistent units for both concentration and volume during the use of the calculation and keep in mind that ( $1\mu\text{g/mL} = 1\text{mg/L} = 1000\mu\text{g/L}$ ,  $1\text{L} = 1000\text{mL} = 1000000\mu\text{L}$ , and  $1\text{mL} = 1000\mu\text{L}$ )

- 9.10 Percent Relative Intensity (%RI) for internal standard assessment (ISTD):

$$\%RI = \text{Intensity of ISTD}_{\text{sample}} / \text{Intensity of ISTD}_{\text{CalBlk}} * 100\%$$

- 9.11 Relative Standard Error (RSE – expressed as a percentage)

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[ \frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

where:

$x'_i$  = Measured amount of analyte at the calibration level  $i$ , in mass or concentration units

$x_i$  = True amount of analyte at calibration level  $i$ , in mass or concentration units

$p$  = Number of terms in the fitting equation (average – 1, linear = 2, quadratic = 3)

- 9.12 See the current Quality Assurance Manual for other equations associated with common calculations.

## 10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications*, before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

- 10.1.1 Prior to using Method 6010D for quantitation of samples, an initial demonstration of performance packet must be completed. This packet must document:

- The selection criteria for background correction points
- Analytical dynamic ranges including the applicable equations and upper limits of ranges




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- IDLs and Method LLOQs
  - The determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. The data must be kept on file and available for review by the data user or auditor.
- 10.2 Use Prep Data to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
- 10.3 Batch Analyses
- 10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1-20 samples as defined in Chapter 1 of SW-846 and in section 9.3.1 of EPA 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information is maintained in Prep Data computer program.
- 10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration (when analyzing EPA 6010C, 6010D and DOD only) 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) at the conclusion of the sequence (when analyzing EPA 6010C only), 1 Continuing Calibration Blank (CCB) following each CCV, 1 Interference Check Sample A (ICSA), 1 Interference Check Sample AB (ICSAB), Interference Check Sample 2, 1 Lanthanum Interference Check Sample, and 1 Cerium Interference Check Sample following each initial calibration. A (ICSA) and 1 Interference Check Sample AB (ICSAB) at the end of the sequence or at least twice per each eight (8) hour shift. All batch information is maintained in Prep Data computer program.
- 10.4 Supporting Analytical Studies
- 10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 10 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.
- IDLs must be verified quarterly<sup>14,13</sup> or when major instrumentation change occurs.
- 10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICP instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.
- To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the




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analysis of a minimum of three, but preferably five, different increasing concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be verified semi-annually<sup>14.1</sup> or when major instrumentation change occurs.

Method 6010D - LDR standards must be ran daily within ten percent of true value, or dilute all samples above the high standard in the curve.

**STATE NOTE:** For work performed in support of the NC Department of Natural Resources (15A NCAC 02H.0805(a)(7)(I)) for target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

- 10.4.3 Method Detection Limits – See also ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs. Method detection limit studies are performed on blank matrices most closely matching field sample matrices.

- 10.4.4 Inter-element Correction Factors – All inter-element spectral correction factors must be verified and updated every six months or when major instrumentation change occurs.<sup>14.1,14.5,</sup>

Criteria for determining an inter-element spectral interference is an apparent positive or negative concentration of an analyte that is outside the 3-sigma control limits of the calibration blank for the analyte. See Attachment II for a listing of potential interfering analytes and their contributions from SW-846 method EPA 6010B. Testing is performed using 100mg/L single element solutions; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

Suggested analytes that are known to commonly interfere include: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn.






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10.4.5 Proficiency Testing (PT) – See also ENV-SOP-MTJL-0022, *Proficiency Testing Program*. Proficiency testing is performed in the metals department in support of both environmental and industrial hygiene analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

10.5 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and three standards for each target analyte. If the correlation coefficient does not meet the acceptance criteria, see the corrective action guidance listed in Section 11.1.

**NOTE:** For EPA Methods 200.7 & 6010B/D, the linear regression correlation coefficient for the each analyte in the calibration curve lines must be  $\geq 0.995$ .

**NOTE:** For Method 6010D - If a multipoint calibration is used the low standard must be at or below the LLOQ.

**NOTE:** For EPA Method 6010C, the regression correlation coefficient must be  $\geq 0.998$ .

10.6 Initial Calibration Verification (ICV/ICVLL) - Verify the accuracy of the initial instrument standardization by analyzing appropriate check standards following calibration. The routine mid-level ICV must be prepared from a source that is independent of the stock standard used for the preparation of the initial calibration curve. For EPA Method 6010C, a low-level ICV (ICVLL) is also performed as required; however the low level ICV is not required to be from a second source. It may be made from the same stock standard as the calibration standards as long as the initial calibration is verified by a second source in the mid-level ICV.

10.6.1 **EPA Method 6010B/D** - The routine ICV standard recovery results must be  $\pm 10\%$  of the true value for EPA method 6010B/D. The RSD must be  $< 5\%$  for the triplicate passes of the spectrometer. If the RSD exceeds 5% and/or the recovery exceeds 10%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.2 **EPA Method 200.7** - The routine ICV standard recovery results must be  $\pm 5\%$  of the true value for EPA method 200.7. The RSD must be within 3% for the four replicate passes of the spectrometer. If the RSD exceeds 3% and/or the recovery exceeds 5%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.3 **EPA Method 6010C** - The routine ICV standard recovery results must be  $\pm 10\%$  of the true value for EPA methods 6010C. The RSD must be  $< 5\%$  for the triplicate passes of the spectrometer. The ICVLL standard recovery results should be within 70-130%. If the recovery does not meet the criteria for either level of ICV and/or the %RSD is exceeded, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.4 **EPA Method 6010D Low-level Readback or Verification** - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.




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10.6.5 **EPA Method 6010D** Mid-level Readback or Verification - For a multi-point calibration, the midlevel standard should quantitate to within 90-110% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the midpoint of the linear range is analyzed and should recover within 90-110% of the true value.

10.7 Continuing Calibration Verification (CCV/CCVLL) - Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Verification is achieved by analyzing both a CCV standard and a CCB (instrument blank). Continuing calibration verification standards can be created from either primary or secondary source standards from those used in instrument calibration. Continuing instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria per the method being analyzed:

10.7.1 **For SW-846 Method 6010B/D** – Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within  $\pm 10\%$ . The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within  $\pm 50\%$ .

10.7.2 **For EPA Method 200.7** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within  $\pm 10\%$ . The RSD must be within 3% for the triplicate passes of the spectrometer.

10.7.3 **For SW-846 Method 6010C** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within  $\pm 10\%$ . The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within  $\pm 30\%$ . ICVLL are analyzed at the beginning of the analytical run and CCVLL are analyzed at the end of the analytical run for 6010C.

10.8 Method/Calibration/Rinse Blanks

10.8.1 Method Blank

10.8.1.1 A method blank is generated for each analytical batch during sample preparation to determine if any contamination is introduced during sample processing. A method blank is routinely a volume of reagent water that is carried through the entire digestion and analysis procedure with the samples.

10.8.1.2 The method blank must not contain analytes >MDL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium. If target analytes are present in the method blank, corrective action must be taken. See section 11.5 for corrective actions.

**NOTE:** Per DoD QSM, version 5.0, Section 1.7.4.1, DoD/DOE require that method blanks be evaluated to  $\frac{1}{2}$  RL (LOQ) for target analytes and RL (LOQ) for common laboratory contaminants. If contaminants are present in the blank above this level,




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samples must be re-prepared and re-analyzed or reported with appropriate qualification.

**NOTE:** Method 6010D – The method blank is considered to be acceptable if target analyte concentrations are less than  $\frac{1}{2}$  the LLOQ or are less than project-specific requirements.

**State Note:** For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

**State Note:** For West Virginia samples analyzed by Method 6010D, blanks are generally considered to be acceptable if target analyte concentrations are less than  $\frac{1}{2}$  the lower limit of quantitation (LLOQ) or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are  $\geq 10X$  the blank). Other criteria may be used depending on the needs of the project.

For West Virginia samples analyzed by Method 200.7, blank values that exceed the MDL indicate laboratory or reagent contamination should be suspected.

## 10.8.2 Calibration Blank

10.8.2.1 An initial calibration blank is generated for each analytical sequence using acidified reagent water. The CALBLK analyzed prior to the initial calibration standards is used to establish a baseline for the instrument prior to calibration. Great care is required during this analysis to ensure that the baseline is correctly established prior to the calibration of the instrument and the analysis of field samples. Inaccurate baselines established with contaminated calibration blanks degrade precision and accuracy of the analyses performed by creating biases in target analyte calibration.

If the initial calibration blank is grossly negative for a target analyte, then the quantitation of that target analyte in the calibration standards will be biased high due to over compensation by the instrument. This will lead to low recovery issues with the CCV, ICV, field samples, and batch QC samples. If target analytes are present in the CALBLK leading the instrument to make an over correction of the baseline for these targets, then the calibration curve will be biased high yielding a low bias for those target analytes in the CCV, ICV, field samples, and batch QC.

10.8.2.2 Continuing calibration blank (ICB/CCB) is also analyzed following each initial and continuing calibration verification standard within an instrument sequence to verify instrument stability and system cleanliness. This does not baseline correct the instrument for possible contaminants in the background and must be evaluated to ensure that background corrections are appropriate and consistently applied throughout the sequence.



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The ICB must not contain analytes > ½ RL, for CCB must not contain analytes >RL for all 6010 methods or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium.

**NOTE:** West Virginia requires all blank results to be less than the MDL. If the concentration of the target analyte is above the MDL, then corrective action and reanalysis is required, or the data must be qualified as including a potential high bias.

### 10.8.3 Rinse Blank

10.8.3.1 A rinse blank is utilized by the ICP to cleanse the system following the intake of each digestate analyzed. No data is obtained during this rinse and no applicable controls are required for this type of blank. This is merely cleansing the lines throughout the analytical system.

- 10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - A matrix/matrix spike duplicate must be prepared for each matrix for each batch of 10 samples for method 200.7 or 20 samples for method 6010B/C/D, where sufficient sample volume was submitted by the client. Matrix spike and matrix spike duplicate are prepared from a sample aliquot spiked with the known concentration of analytes.

The matrix spike recoveries must meet the criteria in the table below unless the analyte concentration in the sample is at least four (4) times greater than the spike concentration.

Method	Acceptance Criteria	
	Water	Soil
6010B, C, and D	75 – 125%	75 – 125%
200.7	70-130%	NA

Assess that the matrix spike duplicate precision (%RPD) results meet project-established goals acceptance criteria. If no project goals are specified, then results for the RPD must be less than 20%.

- 10.10 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - An LCS/LCSD pair must be digested and analyzed with each batch of 20 samples.

10.10.1 **For SW-846 Method 6010B, 6010C, and 6010D** – The LCS recovery must be 80-120%. The RPD must be less than 20%. When using a certified solid reference material for paint chip analysis, the manufacturer's established limits are used for control limits

10.10.2 **For EPA Method 200.7** – The LCS recovery must be 85-115%. The RPD must be less than 20%.

- 10.11 Interference Check Standards (ICSA/ICSAB/ICSA2/LA/CE) – The ICSA2, LA and CE must be analyzed at the beginning of each analytical run. The ICSA and ICSAB must be analyzed at the beginning and ending of each sequence or twice within each eight (8) hour shift, whichever is more frequent. The recovery of the ICSA and ICSAB elements must be






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80-120%. If the results are unsatisfactory, see section 11.10 for further guidance. Do not proceed until this criterion is met.

**NOTE:** The unspiked elements in the ICSA, ICSA2, LA, and CE are not evaluated by the data capture software. The instrument analyst evaluates whether the unspiked elements are  $\pm$ LLOQ for the ICSA, ICSA2, LA, and CE.

10.11.1 For Method 6010D two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup, and periodically (at least once every six (6) months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that and interference corrections applied are still valid.

10.11.1.1 Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 20mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 10mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 100mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 10mg/L.

The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be  $\pm$ LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements.

The single element SIC checks are performed when the instrument is setup and periodically (at least once every six months) thereafter.

10.11.1.2 Mixed element interference check - The mixed element SIC solution (see section 7.10.3.2) is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than  $\pm$ LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions (see Section 7.10.3.1). These may be present up to the concentration documented plus the LLOQ.

10.12 Serial Dilution (SD) - If the analyte concentration is sufficiently high (minimally 10X the IDL), an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, see section 11.11 for further guidance.




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**For Method 6010D** - If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within  $\pm 20\%$  of the original determination.

- 10.13 Post digestion Spike (PS) - An analyte spike added to a portion of a prepared sample, or its dilution and must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL. If the spike is not recovered within the specified limits, see section 11.11 for further guidance.

**For Methods 6010C and 6010D** – The Post Digestion Spike must recover within  $\pm 20\%$  of the known value.

- 10.14 Internal Standard (ISTD) – Verify the internal standard responses. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample must fall within 60-140% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, see sections 9.15 & 11.9 for further guidance.
- 10.15 Sample Dilution - If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), the sample must be diluted with acidified reagent water and re-analyzed.
- 10.16 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6010D, the LLOQ is initially verified by the analysis of at least seven (7) replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be  $\pm 35\%$  of the true value and RSD should be  $< 20\%$ . In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.
- 10.16.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

## 11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.




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- 11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review*.
- 11.3 Initial Calibration – After analyzing the calibration standards, the curve is reviewed to ensure the acceptance criteria described in section 10.5 are met. If analytes do not meet this requirement, corrective action must be taken. Corrective actions may include re-calibrating the instrument, replacing the tubing on the peristaltic pump, examining blanks and standards for degradation/contamination, or performing instrument maintenance. If the internal standard responses in the calibration standards do not meet the criteria in section 10.14, re-calibrate the instrument.
- 11.4 Initial Calibration Verification (ICV) – If the criteria described in section 10.6 are not met for a target analyte, re-analyze the ICV/ICVLL. If this fails a second time, corrective action must be taken. Re-calibrate and re-analyze the ICV/ICVLL using the same standard. If acceptance criteria are still not met, re-check standard curve and ICV/ICVLL preparation and/or perform routine instrument maintenance. If still not acceptable, refer to manufacturer's instruction or call service representative.
- 11.5 Continuing Calibration Verification (CCV) – The continuing calibration verification standard must agree with the criteria in section 10.7 or the CCV must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action may require re-calibrate the instrument and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If the CCV still does not pass, refer to the manufacturer's instruction or call a service representative.
- 11.6 Blanks – Evaluate the blanks. The analyst must confirm that both the method blanks and the continuing calibration blanks were analyzed at the required frequency. Other items to check are as follows:
  - 11.6.1 The instrument blank or continuing calibration blank (ICB/CCB) must meet the criteria in section 10.8. Corrective actions for method blank contamination include re-prepping the entire batch of samples, or if the site-specific requirements can be met, an elevated detection limit may be used, or the sample data qualified with a "B" qualifier and footnoted.
 

**State Note:** For West Virginia samples analyzed by Method 6010D, if the method blank fails to meet the necessary acceptance criteria, it should be re-analyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed, along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification. For West Virginia samples analyzed by Method 200.7, when blank values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of




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contamination has been corrected and acceptable LRB values have been obtained.

**NOTE:** Method 6010D - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.

11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – Assess that LCS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.10, rinse the instrument and re-analyze. If the LCS/LCSD fails for a second time, re-prepared all samples prepared in conjunction with the failing LCS. The affected samples must be re-digested and re-analyzed along with a new LCS. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client.

11.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – Assess that MS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.9, review the post digestion spike results for similar failures. Review the data to see if similar results are also present in the LCS/LCSD. If the LCS/LCSD are acceptable, the MS/MSD failures can be attributed to matrix interferences and the data can be qualified with a J4 and reported. If similar results are seen in the LCS, then re-prepared all samples prepared in conjunction with the failing MS/MSD. In this case, the affected samples must be re-digested and re-analyzed along with a new MS/MSD pair. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client. If insufficient field sample remains for re-analysis, report results with a J3 and an L3.

**NOTE:** If the sample concentration for an analyte is greater than four times (4x) the spike concentration, a “V” qualifier is used. The “V” qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

11.9 ISTD – The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.14. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution due to a possible interference from the matrix on the ISTD. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.






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- 11.10 Interference Check Standards (ICSA/ICSAB/ICSA2/LA/CE) - Evaluate the ICSA, ICSAB, ICSA2, LA, and CE. The analyst must verify that the ICS checks have been analyzed at the required frequency. If the criteria in section 10.11 are not met, check the background correction protocols currently in place for appropriateness. If these are the initial ICS checks run after daily calibration, re-analyze the CALBLANK and re-calibrate the instrument. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-analyze the ICSA and ICSAB. If failure persists, perform instrument maintenance as needed, recalibrate and re-analyze any samples in the previous run that may have been affected.
- 11.11 Serial Dilution/Post-digestion Spike – The analyst must verify that the SD and PS have been analyzed at the required frequency. If either of these tests fails to meet the required criteria in sections 10.12 & 10.13, the possibility of a matrix interferent should be suspected. An O1 qualifier is used when either sample type fails due to matrix interferences.
- 11.11.1 Serial Dilution - An analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.
- 11.11.2 Post-digestion Spike - The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.
- CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.
- 11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.12.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.
- 11.12.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.
- 11.12.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.
- 11.12.4 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J+ qualifier indicating the high bias with no impact on the field sample analysis due to the bias present.
- 11.12.5 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.
- 11.12.6 Sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.




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11.12.7 Samples with multiple elements above the LDR must be diluted to verify interferences are not present

**STATE NOTE:** Drinking water samples analyzed using this procedure for compliance cannot be qualified.

## 12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that a laboratory waste management practice be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051, *Waste Management Plan*.

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

## 13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.

13.4 In the May 2012 Methods Update Rule, the EPA revised the previous interpretation of EPA 200.7 to include the use of axial torch orientation in the published method. Either axial or radial orientation is acceptable.

## 14.0 REFERENCES

14.1 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010B, Revision 2, December 1996.

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

14.3 *Identification of Test Procedures*, 40 CFR §136.3.

14.4 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR §141.23.

14.5 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010C, Revision 3, February, 2007.

14.6 *Hardness by Calculation*, Standard Methods 2340B, 20<sup>th</sup> Edition.

14.7 *Hardness by Calculation*, Standard Methods 2340B, 2011.

14.8 *Hardness by Calculation*, Standard Methods 2340B, 1997.

14.9 *Inorganic Analytes*, SW-846 Chapter 3, Revision 4, February, 2007.

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**Attachment I: Revision History****Current Version (Pace National):**

Date	Description of Revisions
8/13/2020	Technical and quality review and update. Revised header. Revised sections 2.7, 7.5, 7.11, 8.1.7, 10.3.2, 10.11, 11.8 and 11.10. Added sections 2.7.3, 2.7.4, 2.7.5, 7.11.3, 7.11.4, 7.11.5 and 7.12 and renumbered as necessary. Revised all subsections of 3.0.

**Superseded Versions (ESC Lab Sciences SOP#340386):**

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.
16	6/27/14	Complete Rewrite and update.
17	12/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.13.1, 2.13.1, 2.23, 5.1.3, 6.1.1, 6.1.2, 7.1, 7.6, 7.7, 7.8, 7.9, 7.10.1, 7.10.2, 7.11, 7.12, 7.13, 7.14, 7.16, 8.1.3, 8.1.5, 8.1.11, 9.4, 10.2, 10.3.1, 10.3.2, 10.4.2, 10.4.4, 10.9, 10.10, 10.14, 11.12.6, and 12.2. Revised Tables 1.2a, and 1.10, Deleted Sections 2.22 and 7.12. Added Attachment III.



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**TITLE:** Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

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Version	Date	Description of Revisions
18	10/28/2016	Technical and quality review and update. Update per South Carolina DHEC correspondence of 6/24/16. Header and signature block re-formatting. Revised SOP title. Revised Sections 1.1, Table 1.2a, 1.3, 1.6, 1.10, 2.12, 2.15, 5.6, 7.5.2, 7.10.1, 9.1, 10.2, 10.3.1, 10.4.2, 10.4.3, 10.4.5, 10.5, 10.6.1, 10.6.2, 10.7.1, 10.8.1.2, 10.8.2.2, 10.10.1, 10.16, and Attachment III Table 2. Deleted Table 1.2b. Deleted Sections 2.2, 2.12, 2.13, 2.14, 2.15, 2.17, 2.20, through 2.33, 3.5, 4.8, 7.14, 7.16, 8.1.3, 9.7 through 9.10, 9.13, 10.4.5.1, 10.4.5.2, 10.4.5.3, 10.10.3, 13.5, 14.7, 14.8, 14.9, and 14.10. Added Sections 2.14, 7.10.3 and all subsections, 9.11, 9.12, 10.1.1, 10.6.4, 10.6.5, 10.8.1.2, 10.9.1 and all subsections, 10.11.1 and all subsections, and 11.6.1.
19	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Sections 1.5, 3.1, 7.9, 10.11, 14.1, 14.2, 14.3, 14.4, 14.5, 14.6, 14.7, 14.8, 14.9, and Attachment III Table 5.
20	7/27/18	Update in response to WI and AZ audit findings. Changed logo and references to “ESC” to “Pace National”. Revised Section 7.14, 10.4.4, and 10.7.3. Added State Note to Sections 10.8.1.2 and 10.11. Also added 6010C note to Section 10.13
21	9/10/18	Update in response AZ audit finding CAR3296. Revised Post-Spike acceptance criteria to $\pm 20\%$ in Section 10.9.1.2

### Superseded Versions (Pace National):

Date	Description of Revisions
1/27/2019	Technical and quality review and update. Deleted header, footer and signature. Revised sections 1.0, 1.3, 1.5, 1.6, 1.7, 1.13, 1.13.1, 2.8.1(2.7.1), 2.8.2(2.7.1), 4.2, 4.4, 4.7, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5.1, 7.5.3, 7.6, 7.7, 7.8, 7.9, 7.10, 7.10.1, 7.10.2, 7.13, 9.5, 9.6, 9.9, 10.1, 10.2, 10.3, 10.4, 10.4.3, 10.4.5, 10.5, 10.6.3, 10.6.4, 10.6.5, 10.7.3, 10.8, 10.8.1, 10.8.1.2, 10.8.2, 10.8.3, 10.10.1, 10.11, 10.11.1.2, 10.12, 10.13, 10.16, 11.1, 11.2, 11.12.6, 12.1, 12.2, 14.2, 14.6, 14.7 and 14.8. Revised Table 1.10. Added section 7.5 and renumbered as necessary. Deleted sections 7.10.3, 7.10.3.1, 7.10.3.2, 7.11, 7.12, 7.14, 10.9.1, 10.9.1.1 and 10.9.1.2. Revised Attachment I. Revised Attachment II sections 4.10, 4.17 and 4.18.
5/7/2019	Technical and quality review and update. Revised sections 1.1, 1.2, 2.14, 8.1.1.2, 10.3.2, 10.9, 10.11, 10.11.1, 10.11.1.1, 10.11.1.2, 11.8, 11.12.6 and 12.1. Correctly numbered section 7.6.1.1.
6/19/2019	Added corporate header and footer. Revised based on LA DW Auditor's comments. Revised Sections 8.1.6, 10.3.2, and Attachment I.
10/16/2019	Revised section 1.2 and 10.7.1. Added section 11.12.7.
2/4/2020	West Virginia audit response. Revised Section 10.8.2.2.
4/8/2020	Revision in response to West Virginia audit deficiency. Revised Sections 10.8.1.2 and 11.6.1.





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### Attachment II: Potential ICP interferences arising from analytes present in field samples at concentrations of 100mg/L

Analyte	Wavelength (nm)	Interferant <sup>a,b</sup>									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

<sup>a</sup> Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Mg - 1000 mg/L
Ca - 1000 mg/L	Mn - 200 mg/L
Cr - 200 mg/L	Ti - 200 mg/L
Cu - 200 mg/L	V - 200 mg/L
Fe - 1000 mg/L	

<sup>b</sup> The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

<sup>c</sup> Interferences will be affected by background choice and other interferences may be present.

**NOTE:** Using the above table, if analyzing for Lead in a sample containing 1000mg/L Aluminum, the lead results could demonstrate a high bias of 0.17mg/L. (If the sample contained 10000mg/L of Al, the bias in lead could be 1.7mg/L), if background corrections are not accurately applied by the instrument.



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### Attachment III: DoD Requirements

#### 1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing pump tubing, replacing the torch, cleaning the nebulizer, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

#### 2.0 Computer Hardware and Software

QTegra, Version 2.4

#### 3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
Poor Precision	Nebulizer Pressure	Pressure should be about 0.15 mPa for aqueous solutions. If pressure is substantially higher, clean the nebulizer orifice or replace it entirely.
	Pooling in Spray Chamber	Usually caused by an oily film in the spray chamber. Aspirate 0.1% HF solution for about 20 seconds or 0.01% Triton X-100 solution.
	Center Tube	Replace the tube.
	Capillary Tubing	Air bubble migration through tubing should be smooth and consistent. Replace kinked/pinched tubing.
	Peristaltic Pump	Adjust platen pressure. Check for leaks. Replace damaged pump.
Poor Accuracy	Pump Rate	Ensure the flush pump rate is the same as the analysis pump rate.
	Flush Time	Ensure proper time set for adequate rinse (typically 30 seconds).
Poor Detection Limits	Dirty Window or Mirror	Clean or replace dirty components.

#### 4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction



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factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$ , whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$ , whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: $0^{\circ}\text{C}$ to $6^{\circ}\text{C}$ Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually  Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use  Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature

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**Table 2. Support Equipment Checks**

Performance Check	Frequency	Acceptance Criteria
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g.,  $1.00 \pm 0.01\text{g}$ ) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly  $1.00\text{g} \pm 0.01\text{g}$ , as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
  - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
  - B Blank contamination. The recorded result is associated with a contaminated blank.
  - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
  - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).
- Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).
- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a






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measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.

- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
  - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
  - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
  - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
  - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
  - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.




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- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.19 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
  - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
  - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.



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**Table 3. LCS Control Limits – Method 6010 Solid Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

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**Table 4. LCS Control Limits – Method 6010 Water Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115



**STANDARD OPERATING PROCEDURE**

**TITLE:** ENV-SOP-MTJL-0215 Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$ .	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily	All reported analytes within $\pm 20\%$ of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). Low-level calibration check standard should be less than or equal to the LOQ.  If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< \frac{1}{10}$ th the amount measured in any sample or $\frac{1}{10}$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results $>10X$ the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< \frac{1}{10}$ th the amount measured in any sample.	ICB: Correct problem and repeat ICB/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results $>10X$ the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm$ 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply 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 shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations $> 50 \times$ LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations $< 50 \times$ LOQ prior to dilution.

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**Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

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## Document Information

<b>Document Number:</b> ENV-SOP-MTJL-0163	<b>Revision:</b> 06
<b>Document Title:</b> pH, Manual or Automated (EPA Methods 150.1, 9040B, 9040C, 9045C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids using these Methods	
<b>Department(s):</b> Wet Chemistry	

## Date Information

<b>Effective Date:</b> 17 May 2020
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## Notes

<b>Document Notes:</b>
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All Dates and Times are listed in: Central Time Zone



**Signature Manifest****Document Number:** ENV-SOP-MTJL-0163**Revision:** 06**Title:** pH, Manual or Automated (EPA Methods 150.1, 9040B, 9040C, 9045C and 9045D; SM 4500H+ B)  
Including Corrosivity for Solids and Liquids using these Methods

All dates and times are in Central Time Zone.

**ENV-SOP-MTJL-0163****QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Regina Stone (006490)	Quality Analyst 3	11 May 2020, 10:30:26 AM	Approved

**Management Approval**

Name/Signature	Title	Date	Meaning/Reason
Keith Blanchard (004545)	Manager	11 May 2020, 07:48:55 AM	Approved
James Burns (006456)	Manager - EHS	11 May 2020, 08:56:18 AM	Approved
Heidi Ferrell (006481)	Manager - Operations	11 May 2020, 01:24:41 PM	Approved




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## STANDARD OPERATING PROCEDURE

**TITLE:** ENV-SOP-MTJL-0163 pH, Manual or Automated (EPA Methods 150.1, 9040B, 9040C, 9045C and 9045D; SM 4500 H+B) Including Corrosivity for Solids and Liquids Using These Methods

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### 1.0 SCOPE AND APPLICATION

- 1.1 This procedure is for use only by, or under the supervision of, trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. EPA Methods 9040B and 9040C are used to measure the pH of aqueous liquids and those multiphase wastes with an aqueous phase that is at least 20% of the total volume of sample. EPA Methods 9045C and 9045D are used to measure the pH of solids, sludge, or non-aqueous liquids. If water is present, its volume must be less than 20% of the total sample volume. SM4500H<sup>+</sup> B is used to measure the pH of drinking, surface, and saline waters, acid rain, and domestic and industrial wastes. EPA 150.1 can be used to measure the pH of drinking water. See section 13.3 for limitations.
- 1.2 Corrosivity is designed to identify wastes posing hazards to human health or environment.
  - 1.2.1 The corrosivity characteristic is designed to identify wastes that might pose a hazard to human health or the environment due to their ability to:
    - 1.2.1.1 Mobilize toxic metals if discharged in a landfill environment.
    - 1.2.1.2 Corrode handling storage, transportation, and management equipment.
    - 1.2.1.3 Destroy human or animal tissue in the event of inadvertent contact.
  - 1.2.2 EPA has selected two properties upon which to base the definition of a corrosive waste. These properties are pH and the corrosivity toward Type SAE 1020 steel<sup>14.6</sup>.
- 1.3 Corrosivity by Langelier Saturation Index (LSI) is calculated using the results from Total Dissolved Solids (TDS), Temperature, Calcium Hardness (calculated from total calcium), and Total Alkalinity.

### 2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The pH of samples is determined electrometrically using a combination electrode that incorporates both measuring and reference electrode. Automatic temperature compensation is utilized to compensate for temperature variations during analysis. Both electrodes, in combination with a pH meter that can interpret the electrode responses, are calibrated using a series of standards having known pH values that bracket the range of samples. Aqueous samples are measured directly. Non-aqueous samples require extraction with reagent water prior to analysis. Corrosivity determination is made using the pH value obtained. A pH value of <2, or ≥12.5 is determined to be corrosive<sup>14.6</sup>. Values >2 and <12.5 are determined to be non-corrosive by this procedure.
- 2.2 pH - the inverse logarithm (base 10) of the hydrogen ion activity.
- 2.3 Corrosivity – The ability of a substance to destroy and/or damage other substances with which it comes into contact. Routinely, common corrosives are either strong acids/bases or concentrated solutions of certain weak acids/bases.
- 2.4 Langelier Saturation Index (LSI) – An indicator that the water is corrosive or scale-forming.
- 2.5 See the current Quality Assurance Manual for other definitions associated with terms found in this document.




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### 3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each chemical or sample being diluted in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- 3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.
- 3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.
- 3.4 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.
- 3.5 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

### 4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Aqueous Samples should be analyzed as soon as possible, preferably in the field at time of sampling. The Federal Register citation 40 CFR 136.3 (Table II) specifies a maximum holding time of 15 minutes. Samples that are shipped to the laboratory are reported with a T8 qualifier. Non-aqueous samples should be analyzed as soon as the preparation is completed. The results should also be reported with a T8 qualifier. In all cases, efforts are made to analyze within 24 hours of collection.
- 4.3 Some samples are subject to changes in pH when exposed to the atmosphere. Sample containers should be filled completely and kept sealed prior to analysis.
- 4.4 Store samples during shipping and prior to analysis at  $4^{\circ} \pm 2^{\circ}\text{C}$ . Allow samples to reach ambient temperature prior to analysis.

### 5.0 INTERFERENCES

- 5.1 Oily material or particulate matter may coat electrodes and impair responses. Remove these interferences by gently washing with detergent, rinsing with reagent water, and wiping with a soft cloth.




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- 5.2 Electrode response can differ at varying temperatures. Using a temperature-compensating electrode and a pH meter with temperature compensation capability controls fluctuations of this kind.
- 5.3 In some samples, pH change is inherent at different temperatures. This error is sample dependent and cannot be controlled. Both pH and temperature, at the time of analysis, should be noted on the report.
- 5.4 Acid rain samples must not be analyzed using a magnetic stirrer because pH values will change if the sample is not in equilibrium with the air. Instead, swirl the sample gently.
- 5.5 Sodium concentration in samples at a pH value greater than 10 can cause a bias in pH measurements. Low Sodium Error” electrodes are used.
- 5.6 Scratches, deterioration, and accumulation of debris on the glass surface are the most common causes of electrode failure. Use, store and maintain electrodes according to manufacturer's instructions.
- 5.7 Organic solvents are damaging the electrode. If a sample is an organic solvent, do not allow it to contact the electrode.

## 6.0 EQUIPMENT AND SUPPLIES

- 6.1 Instruments:
  - 6.1.1 Manual: pH meter with temperature compensation capability (Thermo Scientific Orion Versa Star or equivalent).
  - Combination pH electrode (Orion 8157BNUMD pH/ATC Triode or equivalent)
  - Temperature-compensating electrode (Thermo Scientific ATC or equivalent)
  - Magnetic stir plate (Thermo SP-131015Q or equivalent)
  - Magnetic stir bars (Fisher 14-511-58 or equivalent)
  - NOTE: Electrodes must be low-sodium type.
- 6.2 Stirring Hot Plate (Thermo SP-131015Q or equivalent)
- 6.3 Centrifuge tubes (Corning 430829 or equivalent)
- 6.4 Top loading analytical balance capable of weighing to 0.01g (Ohaus Scout Pro or equivalent)
- 6.5 Disposable Beakers (Fisher 01-291-10 or equivalent)
- 6.6 Centrifuge (Thermo Fisher Sorvall ST40 or equivalent)

## 7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system.
- 7.2 pH 1 buffer (Ricca 1489-32 or equivalent)






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- 7.3 pH 4 buffer (Inorganic Ventures PHRED- 4 or equivalent)
- 7.4 pH 7 buffer (Inorganic Ventures PHYellow-7 or equivalent), used for ICV and CCV, but must be from a different vendor or lot
- 7.5 pH 10 buffer (Inorganic Ventures PHBLUE-10 or equivalent), used for LCS
- 7.6 pH 13 buffer (Ricca 1625-16 or equivalent)

**NOTE:** Buffers can be used only once i.e. daily. Use the smallest possible aliquots of each buffer that provides sufficient probe immersion and discard remainders of the aliquots when completed. Opened stock buffers are valid for one (1) month but cannot exceed manufacturers expiration date.

## 8.0 PROCEDURE

### 8.1 Matrix Determination

- 8.1.1 Verify that the sample is water soluble by placing a few drops into a test tube containing water. Verify mixing. If two layers are visible, treat sample as an oily waste.

### 8.2 Preparation of non-aqueous samples.

- 8.2.1 Tare a disposable beaker on the top-loader balance.
- 8.2.2 Weigh up to 20 grams of sample into a centrifuge tube or beaker.
- 8.2.3 Add an equal weight of reagent water.
  - 8.2.3.1 If the sample is hygroscopic, further dilution is allowed. Record.
- 8.2.4 Mix sample and water by gentle stirring for 5 minutes.
- 8.2.5 Allow solids to settle for one (1) hour (soil) or 15 minutes (oil) to reach equilibrium. If needed, centrifuge (2000 rpm or greater) for about 2 minutes. Decant oil layer or transfer water layer to another vessel.

### 8.3 Manual Sample Analysis

- 8.3.1 Place a stir bar into a disposable beaker and add sufficient sample, buffer, or water extract to cover the sensing unit on the electrode and to allow for adequate clearance between the electrode and the stir bar.
  - 8.3.1.1 For preparation of the LCS, use an aliquot of a second source pH buffer or certified reference material.
- 8.3.2 Place the sample on the stir plate and begin stirring gently in order to generate as little agitation as possible at the air/sample interface. See 5.4 for special handling of acid rain samples. See 5.7 for special handling of organic solvent samples.
- 8.3.3 Immerse the electrodes in the sample.
- 8.3.4 When the reading is stable (less than 0.1 pH unit drift), the “Stable” prompt will appear on the meter display, then record the result as it is displayed Then take a




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second aliquot of sample and perform the analysis on it. All aqueous (water) samples must be analyzed in duplicate. Record.

**Note:** Any sample pH measurement above 12.0 must be re-measured at a temperature reading of 25°C +/- 1.0. Document the exact temperature measurement with these type samples. This is essential.

- 8.3.5 Rinse the electrodes with reagent water and continue analyzing the remainder of samples and buffers in the Workgroup.

## 9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Due to the interrelationship between temperature and pH, both values should be noted on the test report.
- 9.2 For Corrosivity: Report results as either “**corrosive**” (pH ≤2.0 or ≥12.5) or “**not corrosive**” (pH >2.0 - <12.5)<sup>14,6</sup>. Report actual pH values for QC samples (blank, LCS) to allow the review for acceptability.
- 9.3 Calculation of the slope of the calibrated pH meter when pH readings can be provided in millivolts (mV) and slope is not automatically provided by the meter<sup>14,7</sup>:

$$\text{Slope (as \%)} = [\text{mV at pH 7} - \text{mV at pH 4}] \times 100/177$$

**NOTE:** This is not the same as used in linear regression. The slope is relative to theoretical using Nernst equation.

- 9.4 See the current Quality Assurance Manual for other equations associated with common calculations.
- 9.5 Langelier Saturation Index calculation for corrosivity:

$$\text{Langelier Index} = \text{pH} - \text{pH}_s$$

Where:  $\text{pH}_s = (9.3 + A + B) - (C + D)$

$A = (\text{Log}_{10} [\text{TDS}] - 1) / 10$

$B = -13.12 \times \text{Log}_{10} (^\circ\text{C} + 273) + 34.55$

$C = \text{Log}_{10} [\text{Ca}^{2+} \text{ as CaCO}_3] - 0.4$

$D = \text{Log}_{10} [\text{alkalinity as CaCO}_3]$

## 10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing and/or laboratory control sample analysis. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.
- 10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
- 10.3 Batches are defined as sets of 1-20 samples. Batch analysis must include the following: 1 Initial Calibration Verification (ICV), 1 Laboratory Control Sample (LCS), 1 Sample




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Duplicate for every 10 samples (soils), 1 Continuing Calibration Verification (CCV) every 10 analyses and at the end of the sequence.

- 10.4 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV): A CCV standard must be analyzed immediately after calibration as well as at a frequency of every ten analysis (including LCS, duplicates, and blanks in the count) during an analytical sequence. The CCV standard must also be analyzed after the last analytical sample. The measured concentration must be within 0.1su of the true value.
- 10.5 Laboratory Control Sample (LCS): One LCS must be analyzed with every batch of 20 samples. The measured concentration of the soil LCS must fall within the acceptable range provided by the standard manufacturer.
- 10.6 Sample Duplicate: One sample duplicate must be analyzed with every set of 10 samples for non-aqueous samples. All aqueous samples must be analyzed in duplicate as described in section 8.3.4. A sample duplicate is an additional aliquot of the original sample that has been taken through the entire procedure. The results must be within 0.1su of each other for water matrices and the calculated RPD should be  $\leq 1\%$  for solid samples.
- 10.7 Calibration Curve (linearity check): A calibration curve must be performed daily. The slope must be 95-105%<sup>14.7</sup>.
- 10.8 The requirements of ENV-SOP-MTJL-0040, Verification of Automatic Temperature Compensation Probes on Laboratory pH Meters, must be performed quarterly for ATC-equipped pH meters. Results of this verification must be documented on controlled document GEN-01 and archived at H:\QAQC\Public\ATC verifications.

## 11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All data must undergo a primary review by the analyst. The analyst should review sample results and make sure that they have been confirmed. All calculations must be checked. Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.
- 11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
- 11.3 Data that does not meet acceptable QC criteria may be acceptable for use in certain cases.
  - 11.3.1 Due to the "As Soon As Possible" method specified holding time, results for samples analyzed in the laboratory should be reported with a T8 (Sample received out of hold time) qualifier.
  - 11.3.2 Sample duplicate – If the difference between the sample and sample duplicate is greater than  $\pm 0.1$ su, the sample should be reported with a J3 qualifier for precision beyond the range for the method. All samples must be analyzed in duplicate with a second aliquot of sample to perform the duplicate determination.

**NOTE:** Data qualification is not allowed for drinking water samples.

- 11.4 LCS: If the LCS is not within acceptance criteria, the sample(s) may be re-analyzed once. If the failure of the LCS persists, the instrument must be re-calibrated and any needed maintenance performed. All samples analyzed in conjunction with the failure must be re-analyzed.




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11.5 ICV/CCV: If the ICV/CCV are not within acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

11.6 Sample Duplicate: If the results of the sample duplicates exceed the acceptance criteria, prepare another aliquot of the sample and analyze to confirm the initial readings. If the results do not confirm, flag the results with a J3 qualifier.

11.7 Calibration Curve: If the calibration curve does not meet the acceptance criteria, it must be re-analyzed. A calibration curve must be successfully run before sample analysis can be performed. Generally, use at least three buffers at three pH value difference i.e. 4, 7 and 10.

## 12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051, *Waste Management Plan*.

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

## 13.0 METHOD MODIFICATIONS/CLARIFICATIONS

## 14.0 REFERENCES

- 14.1 *pH Value, Electrometric Method*, Standard Methods 4500H<sup>+</sup> B, 20<sup>th</sup> Edition.
- 14.2 *pH Electrometric Measurement*, SW-846 9040C, Revision 3, November 2004.
- 14.3 *Soil and Waste pH*, SW-846 Method 9045C, Revision 3, January 1995.
- 14.4 *Soil and Waste pH*, SW-846 Method 9045D, Revision 4, November 2004.
- 14.5 Accumet AB15 Plus instrument manual.
- 14.6 *pH Value, Electrometric Method*, Standard Methods 4500H<sup>+</sup> B, 2000.
- 14.7 *pH Value, Electrometric Method*, Standard Methods 4500H<sup>+</sup> B, 2011 or on-line version.
- 14.8 Code of Federal Regulations, Title 40, Part 261.22.
- 14.9 *EPA Manual for the Certification of Laboratories Analyzing Drinking Water*, EPA 815-R-05-004, 5<sup>th</sup> Edition, January 2005.
- 14.10 *pH (Electrometric)*, EPA Method 150.1, Approved for NPDES, Editorial Revision 1978, 1982.
- 14.11 *Calcium Carbonate Saturation*, Standard Methods 2330 B, 2011



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**Attachment I: Revision History****Current Version (Pace National):**

Date	Description of Revisions
5/9/2020	Technical and quality review and update. Deleted sections 1.1, 1.2, 1.3, 5.7.1, 6.2, 8.1.1, 8.1.2, 8.1.3, 8.2.6, 11.4, 13.1, 13.2, 13.3 and 13.4 and renumbered as necessary. Revised sections 1.4, 2.1, 2.2, 2.3, 2.4, 4.2, 4.4, 5.1, 5.5, 5.7, 6.1.1, 7.6, 8.2.2, 8.2.3, 8.2.3.1, 8.2.4, 8.2.5, 8.3.1, 8.3.2, 8.3.4, 8.3.5, 9.3, 10.3, 10.4, 10.6, 10.7, 11.7, 11.8 and 14.7. Replaced section 3.0, Health and Safety. Revised Attachment II, sections 4 and 5. Added section 1.11 to Attachment III.

**Superseded Versions (ESC Lab Sciences SOP#340335):**

Version	Date	Description of Revisions
0	11/13/92	Origination
1	8/3/00	
2	8/22/00	
3	1/16/02	
4	7/23/03	
5	1/27/04	
6	1/19/06	
7	9/7/07	Complete Revision for Format, Technical Content and Clarity.
8	5/21/09	Annual Technical and Quality Review. Removed EPA Method 150.1 (MUR); update EPA methods and references; Revised sections 10.4 – 10.8 & 11.0
9	10/01/10	Annual Technical and Quality Review. Added sections 2.8 and 10.4; Revised sections 4.2, 5.7, 7.1, 8.1.3, 8.1.8, 8.2.6, 10.6, 10.7, and 12.1.
10	11/3/11	Annual Technical and Quality Review. Added sections 13.3 and Attachment II; Revised sections 2.8 and 12.1.
11	2/28/12	Annual Technical and Quality Review. Added Attachment III; Revised sections 7.4, 8.1 and Attachment II; Added sections 7.7.
12	5/24/13	Annual Technical and Quality Review. Revised Attachment II and sections 4.2, 6.1, 8.4, and 10.5; Added sections 8.2, 8.5, 9.2, 10.7, 11.8, 14.4 and 14.5; Removed sections 2.4, 6.3 through 6.5 and 10.5. Consolidated 340335 and 340335A into single procedure
13	8/8/14	Annual Technical and Quality Review. Revised Attachment II & III and sections 1.3, 2.1, 6.1, 8.1, 8.3.2, 8.3.5, 8.3.6 and 10.5; Added note in section 7.0 and added sections 1.5, 2.3, 9.3, 9.4, 13.4, 13.5 and 14.6 through 14.8. Consolidated 340335 and 340306 into single procedure
14	10/20/2015	Technical and Quality review and revision. Revised header and signature block. Revised Title and Sections 1.1, 1.2, 5.7.1, 12.2, 13.5, and 14.2.
15	11/4/2016	Technical and quality review and update. Update per SC DHEC correspondence of 10/19/2016. Header and signature bar re-formatting. Revised Sections 2.4, 7.1, 8.3.4, 10.4, 11.6, 14.1, 14.2, 14.6, 14.7, and 14.10. Deleted Sections 2.5 through 2.8, 9.2, and 13.2. Added Sections 6.3, 9.4, 10.8, 14.3, and 14.4.

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Version	Date	Description of Revisions
16	4/9/2018	Technical and quality review and revision. Changed ESC logo. Revised sections 3.1, 4.2, 4.4, 6.1.1, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5, 7.6, 8.3.2, 8.3.4, 10.3, 13.4, 14.1, 14.3, 14.4, 14.6, 14.7, 14.8 and 14.9. Deleted sections 6.1.2, 7.7, 8.5 (and subsections). Deleted sections 8.1 and 8.2 (and subsections) and renumbered as necessary. Deleted section 8.4.1.1 and renumbered next section. Added section 6.7. Revised Attachment II sections 3c and 3e.

**Superseded Versions (Pace National):**

Date	Description of Revisions
3/22/2019	Technical and quality review and revision. Deleted header, footer and signature bar. Revised sections 3.0, 5.3, 7.1, 7.4, 7.5, 8.1.6, 8.2.1.1, 8.2.4, 9.2, 10.1, 10.2, 10.3, 10.4, 10.5, 10.8, 11.2, 11.5, 12.1, 12.2 and 13.4. Revised Attachments I, II and III.
4/21/2019	Technical and quality review and revision. Added sections 1.6, 2.4, 9.5 and 14.11 and renumbered as necessary. Revised sections 7.1, 14.6 and 14.7. Revised Attachment I.
8/16/2019	Revision in response to CAR3681. Added corporate header and footer. Revised Sections 1.3, 4.2, 7.5, 8.2.4, 8.2.5, 10.5, 10.6, 11.3.2, 11.4, 11.7, 11.8, Att II Section 2, Att II Section 4, Att III Sections 1.3, 1.4, 1.5, 1.6, and 1.7. Deleted 13.1. Added 13.4.
9/19/2019	Update in response to SC DHEC8/29/2019 technical review letter. Revised Section 10.4.
2/20/2020	Revision in response to Las Vegas Audit (CAR LV-001). Added Section 8.1 and all subsections. Re-numbered as appropriate.



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**Attachment II: Analytical Tech Notes**

1. Calibrate (Attachment III)
2. Prepare soil samples (See Section 8.1)
3. Run Samples and QC
  - a. ICV (pH = 6.9-7.1)
  - b. LCS ( $\pm 0.1$ su)
  - c. 7 samples plus 1 duplicate ( $\pm 0.1$ su)
  - d. CCV (pH = 6.9-7.1)
  - e. 9 samples plus 1 duplicate ( $\pm 0.1$ su)
  - f. CCV (pH = 6.9-7.1)
4. Do not put probes into organic solvent
5. For Corrosivity Determination: If pH is  $\leq 2$  or  $\geq 12.5$ , report as "corrosive". If pH is  $> 2$  and  $< 12.5$ , report as "not corrosive".




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**Attachment III: Manual Technical Notes:**

1. Calibration – pH calibration procedures vary from meter to meter. The following instructions are used with the Thermo Scientific™ Orion Versa Star pH meter and are provided as general direction. Use of other pH meters in conjunction with this procedure may cause slight variation from the process that follows. Refer to the manufacturer's instructions on calibration and operation of the pH meter in use.
  - 1.1 Press Cal (F1).
  - 1.2 Select desired channel (1 or 2)
  - 1.3 Pour pH 1 buffer into a disposable beaker with a stir bar and place the beaker on a stir plate. Use sufficient volume of solution to cover the sensing unit on the probe and to allow for adequate clearance between the probe and the rotating stir bar. Rinse the electrodes and place them in the buffer solution. When the display prompts that the reading is stable, press [accept] (F2) once. Record the mV and temperature value that is then displayed in the pH calibration log and then proceed with other buffers by selecting [next].
  - 1.4 Place pH 4 buffer on stir plate and repeat instructions from 1.3.
  - 1.5 Place pH 7 buffer on stir plate and repeat instructions from 1.3.
  - 1.6 Place the pH 10 buffer on the stir plate and repeat instructions from 1.3.
  - 1.7 Place the pH 13 buffer on the stir plate and repeat instructions from 1.3.
  - 1.8 When finished select [CAL DONE] button. Record slope in the pH calibration log. Then select [Measure] to begin analyzing samples.
  - 1.9 The slope must be between 95% and 105% for analysis to proceed. If the slope is not within this range, repeat calibration with fresh buffers.
  - 1.10 Buffers can be used only once. Use the smallest possible aliquots and discard.
  - 1.11 Replicate measurements of buffers should be within 0.05su.





## Appendix E - Field Forms

*This roster is required for emergency response planning. All personnel arriving to and from the site must sign this roster. This log does not replace the H&S Orientation*

[illegible]

## TAILGATE SAFETY MEETING REPORT

*A tailgate safety meeting should be held before each unique activity, for each new crew and daily thereafter.*

**Site Name:**

**Date:**

**Contract No.:**

**Delivery Order No.:**

### HEALTH & SAFETY PLAN REVIEW

<input type="checkbox"/> H & S objectives	<input type="checkbox"/> Site emergency procedures	<input type="checkbox"/> Chem. hazards./contaminants	<input type="checkbox"/> Physical hazards
<input type="checkbox"/> Site history	<input type="checkbox"/> Air-horn signals, if any	<input type="checkbox"/> Exposure pathways	<input type="checkbox"/> Well drilling oper.
<input type="checkbox"/> Gen. site hazard assess.	<input type="checkbox"/> Hospital location	<input type="checkbox"/> Exposure monitoring program	<input type="checkbox"/> Noise monitoring
<input type="checkbox"/> Chain of command	<input type="checkbox"/> Telephone location	<input type="checkbox"/> Acute symptoms, if distinct	<input type="checkbox"/> Heat stress
<input type="checkbox"/> Visitor policy	<input type="checkbox"/> Response to media inquiries	<input type="checkbox"/> Decontamination procedures	

### FIELD ACTIVITY

Safety Topics and Suggestions

Personal Protective Equipment (PPE)

Emergency Information and Procedures

Injuries and Accidents Since Previous Meeting

Additional comments

Meeting conducted by \_\_\_\_\_ Title \_\_\_\_\_  
 Signature \_\_\_\_\_ Date/Time \_\_\_\_\_



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**FIELD SAMPLING REPORT (Soil or Sediment)**

LOCID _____			Site Location: Joint Base Charleston Weapons				
Site Name: _____			DATE: _____				
<b>SAMPLE INFORMATION</b>							
MATRIX: Soil Sediment (circle )			SAMPLE ID: _____				
SAMPLING METHOD: _____			DUP./REP. OF: _____				
BEGINNING DEPTH: _____			MATRIX SPIKE/MATRIX SPIKE DUPLICATE				
END DEPTH: _____			YES: _____ NO: _____				
GRAB: _____ COMPOSITE: _____			DATE: _____ TIME: _____				
CONTAINER		PRESERVATIVE/ PREPARATION	EXTRACTION METHOD	ANALYTICAL METHOD	CONSTITUENT DESCRIPTION	ANALYZE FOR? (Y/N)	
SIZE	TYPE #						
<b>NOTABLE OBSERVATIONS</b>							
PID READINGS		SAMPLE CHARACTERISTICS			MISCELLANEOUS		
1st		COLOR: _____					
2nd		ODOR: _____					
		OTHER: _____					
<b>FIELD TESTS</b>							
		:			:		
		:			:		
		:			:		
<b>GENERAL INFORMATION</b>							
WEATHER: SUN/CLEAR___ CLOUDY/RAIN___ WIND DIRECTION _____ TEMPERATURE (°C) _____							
SHIPMENT VIA: FED-X___ HAND DELIVER___ COURIER___ OTHER___							
SHIPPED TO: _____							
COMMENTS: _____							
SAMPLER: _____				OBSERVER: _____			

Waste Inventory Tracking Log						
Drum Identifier	Contents (Site, Matrix, Sample Locations, Level of contamination)	Date Filled	Storage Location	Analytical Results (Y/N)	Properly Disposed (Y/N and Subcontractor)	Manifest and Custody completed and returned (Y/N)
Notes:						

<b>QUALITY ASSURANCE REPORT (QAR) DAILY LOG OF ACTIVITIES</b> For use of this form see ER 1180-1-6;.	1. REPORT NUMBER	
9	3. DATE (YYYYMMDD)	4. CONTRACT NUMBER
	5. CONTRACTOR ( <i>or hired labor</i> )	
	6. WEATHER	
7. CQC CONTROL PHASES ATTENDED AND INSTRUCTION GIVEN		
8. RESULTS OF QA ACTIVITIES AND TESTS, DEFICIENCIES OBSERVED, ACTIONS TAKEN AND CORRECTIVE ACTION OF CONTRACTOR. INCLUDE COMMENT PERTAINING TO CONTRACTORS CQC ACTIVITIES.		
9. VERBAL INSTRUCTION GIVEN TO CONTRACTOR ( <i>Include names, reactions and remarks</i> )		
10. HAS ANYTHING DEVELOPED ON THE WORK, WHICH MIGHT LEAD TO A CHANGE ORDER OR FINDING OF FACT?    YES <input type="checkbox"/> <input type="checkbox"/> NO		



11. INFORMATION ON PROGRESS OF WORK, CAUSES FOR DELAYS AND EXTENT OF DELAYS, WEATHER, PLANT, MATERIAL, ETC.,

12. INFORMATION, INSTRUCTIONS OR ACTIONS TAKEN NOT COVERED ON QCR REPORT OR DISAGREEMENTS.

13. SAFETY: *(Include any infractions of approved safety plan, safety manual or instructions from Government personnel. Specify corrective action taken.)*

14. REMARKS: *(Include visitors to project and miscellaneous remarks pertinent to work.)*

15a. NAME AND TITLE *(Last, First MI)*

b. DATE (YYYYMMDD)

c. QA REPRESENTATIVE'S SIGNATURE

16a. NAME AND TITLE *(Last, First MI)*

b. DATE (YYYYMMDD)

c. SUPERVISOR'S SIGNATURE