



**STUDY AREA PRE-DESIGN INVESTIGATION WORK PLAN
RESIDENTIAL AREAS (OU1, OU2 AND OU5)**

**Study Area
Corning, NY
NYSDEC Project ID 851046**

January 9, 2018

Prepared for:

**Corning Incorporated
Corning, New York**

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Certifications

I Michael H. Corbin certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375 and that this Work Plan was prepared in accordance with all applicable standards and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10).”

Executed on the 9th day of January 2018

Weston Solutions, Inc.

A handwritten signature in black ink, appearing to read "MHC", written over a horizontal line.

(Signature)

Technical Director



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LIST OF ACRONYMS

ASP	Analytical Services Protocol
CAMP	Community Air Monitoring Plan
cfs	cubic feet per second
COPC	constituents of potential concern
CPP	Citizen Participation Plan
DUSR	Data Usability Summary Report
EDD	electronic data deliverable
ELAP	NYSDOH Environmental Laboratory Approval Program
FEMA	Federal Emergency Management Agency
FFS/AA	Focused Feasibility Study/Alternative Analysis
ft amsl	feet above mean sea level
ft bgs	feet below ground surface
GPS	global positioning system
HASP	Health and Safety Plan
IDW	investigative derived waste
in bgs	inches below ground surface
NYCRR	New York Codes, Rules and Regulations
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OU	Operable Unit
PC	public-conservation zoning
PDD	Proposed Decision Document
PDI	Pre-Design Investigation
PID	Photoionization Detector
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
R1	low-density residential zoning
ROW	right-of-way
SCO	soil cleanup objective
SOP	standard operating procedure



LIST OF ACRONYMS (Continued)

SVOC	semi-volatile organic compound
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TCL	Target Compound List
TOGS	Technical & Operational Guidance Series
U.S.	United States
USGS	U.S. Geological Survey
WESTON®	Weston Solutions, Inc.



1. INTRODUCTION

The Study Area is located in the City of Corning, New York, as illustrated on Figure 1-1, and, in general, is bounded by the Chemung River to the south; Post Creek and Interstate 86 to the east and north; and the Guthrie Medical Center, the City of Corning Fire Department, and Centerway to the west. The Study Area is separated into five operable units (OUs) based on location and land use, to assist in advancing properties through the remedial process. The five OUs in the Study Area are identified as follows: the Residential Area (OU1), the Residential Area at the Eastern End of Corning Boulevard (OU2), School/Community Use Areas (OU3), Flood Control Areas (OU4) and the Residential Expansion Area (OU5). The Study Area and OUs are depicted on Figure 1-2.

On June 27, 2014 Corning Incorporated entered into an Order on Consent and Administrative Settlement (June 2014 Order on Consent) with the New York State Department of Environmental Conservation (NYSDEC) to perform characterization activities within a portion of the Study Area¹. From 2014 through 2017, Weston Solutions, Inc. (WESTON®), on behalf of Corning Incorporated, performed characterization activities in OU1 and OU2 under the Study Area Characterization Work Plan dated June 2014, Study Area Work Plan Addendum 1 (Work Plan Addendum 1), Study Area Work Plan Addendum 2 (Work Plan Addendum 2), Study Area Work Plan Addendum 3 (Work Plan Addendum 3) and the Study Area Work Plan Addendum 4) (WESTON, 2014b; WESTON, 2015a; WESTON, 2015b; WESTON, 2017b). Collectively the June 2014 Study Area Characterization Work Plan and its Addenda, as modified, amended and approved by NYSDEC, are referenced herein as the Study Area Work Plan. Sampling activities also were performed by NYSDEC in OU1 and OU2 in 2014 and 2015. In 2015, Arcadis CE, Inc., on behalf of NYSDEC, performed characterization activities in OU5 in 2015 (Arcadis, 2017).

A Focused Feasibility Study/Alternative Analysis (FFS/AA) was prepared by WESTON on behalf of Corning Incorporated to evaluate remedial alternatives for the residential operable units (OU1, OU2 and OU5) contained in the Study Area (WESTON, 2017a). The recommended alternative in the FFS/AA was to excavate up to 2 feet below ground surface (ft bgs) with a cover system.

¹ This portion includes OU1, OU2, OU3 and OU4.



On March 9, 2017, NYSDEC issued for public comment, a Proposed Decision Document (PDD) proposing the recommended alternative of excavation of up to 2 ft bgs with a cover system for properties in OU1, OU2 and OU5 (NYSDEC, 2017a).

On July 12, 2017, NYSDEC issued a final Decision Document (DD) describing the selected remedial alternative of excavation of up to 2 ft bgs with a cover system for properties in OU1, OU2 and OU5 (NYSDEC, 2017b).

Effective December 14, 2017 Corning Incorporated entered into an Order on Consent and Administrative Settlement (December 2017 Order on Consent) with NYSDEC to perform remedial activities and additional characterization activities within the Study Area. This Remedial Action Work Plan (RAWP) has been prepared by WESTON on behalf of Corning Incorporated to perform remedial activities in the residential Operable Units of the Study Area (OU1, OU2 and OU5) described in the NYSDEC DD, pursuant to Section II.A.e of the December 2017 Order on Consent.

1.1 STUDY AREA PRE-DESIGN INVESTIGATION WORK PLAN OBJECTIVES

The purpose of the Pre-Design Investigation (PDI) Work Plan is to gather additional data to assist in defining the extent of Subject Material encountered within OU1, OU2 and OU5. The following materials (collectively referred to as the “Subject Material”) are the subject of the pre-design investigation:

- Soil with concentrations greater than the applicable NYSDEC Soil Cleanup Objectives (SCOs; New York Codes, Rules and Regulations (NYCRR) Subpart 375-6)²
- Layers of fill material containing ash, brick and/or glass³ with concentrations greater than the applicable SCOs; and

² NYSDEC residential SCOs are applicable to the residential properties and NYSDEC restricted-residential SCOs are applicable to the City of Corning rights-of-way.

³ A “layer of fill material containing ash, brick, and/or glass” is defined as a non-native material containing ash, brick, and/or glass with a thickness of greater than 1 inch.



- Layers of fill material containing ash, brick and/or glass that do not contain concentrations greater than the applicable SCOs⁴.

These additional data will help to determine the limits of remediation under the selected remediation alternative of soil excavation up to 2 ft bgs and a soil cover. This PDI Work Plan includes a summary of the characterization sampling activities to date as well as a plan for the pre-design investigation sampling activities in OU1, OU2 and OU5.

The specific objectives of the PDI Work Plan are as follows:

1. Perform additional sampling, as needed, to result in at least four surface soil samples and two soil borings on all residential properties in OU1⁵ and OU5.
2. Perform additional sampling to delineate Subject Material.
3. Perform additional sampling to investigate fill material containing ash, brick and/or glass where analytical results collected to date do not indicate that concentrations of constituents are greater than the SCOs.
4. Perform additional sampling to collect endpoint confirmation samples.

Due to the confidential nature of sample collection on privately-owned residential properties, no sampling location maps are included in the PDI Work Plan. Sample locations are described in tabular form in Section 4.0.

1.2 ORGANIZATION OF THIS DOCUMENT

This PDI Work Plan is organized into the following sections:

- **Section 1 – Introduction.** This section contains an introduction to the project and the objectives of the pre-design investigation activities.

⁴ Layers of fill material containing ash, brick and/or glass which do not contain concentrations greater than the applicable SCOs are being remediated at the direction of NYSDEC. Remediation of this material is not an admission that remediation is necessary and/or that such materials represent a potential for exposure.

⁵ With the exception of 66 properties in OU1 which have been included in Study Area Characterization Work Plan Addendum 4 required by NYSDEC.



- **Section 2 – Background.** This section contains a history of the Study Area, a summary of historical records reviewed to date, and a summary of previous sampling activities performed in OU1, OU2 and OU5.
- **Section 3 – Environmental Setting.** This section contains a brief description of the Study Area location, land use, topography and drainage, geology, hydrogeology, and ecological setting.
- **Section 4 – Pre-Design Remediation Sampling Activities.** This section contains a description of the sampling activities to be conducted, including the locations, types and numbers of samples to be collected, rationale for sample collection, and method of collection for the planned work.
- **Section 5 – Project Management.** This section contains information regarding the scheduling of the pre-design investigation field work, as well as the reporting schedule. Additionally, this section provides details about project logistics, including project controls, management and community relations.
- **Section 6 – References.**

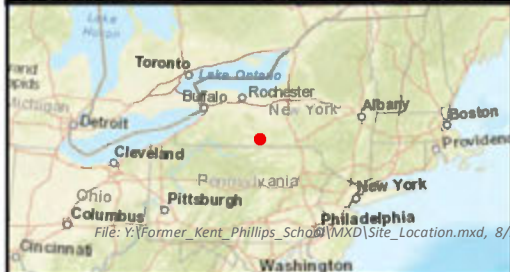
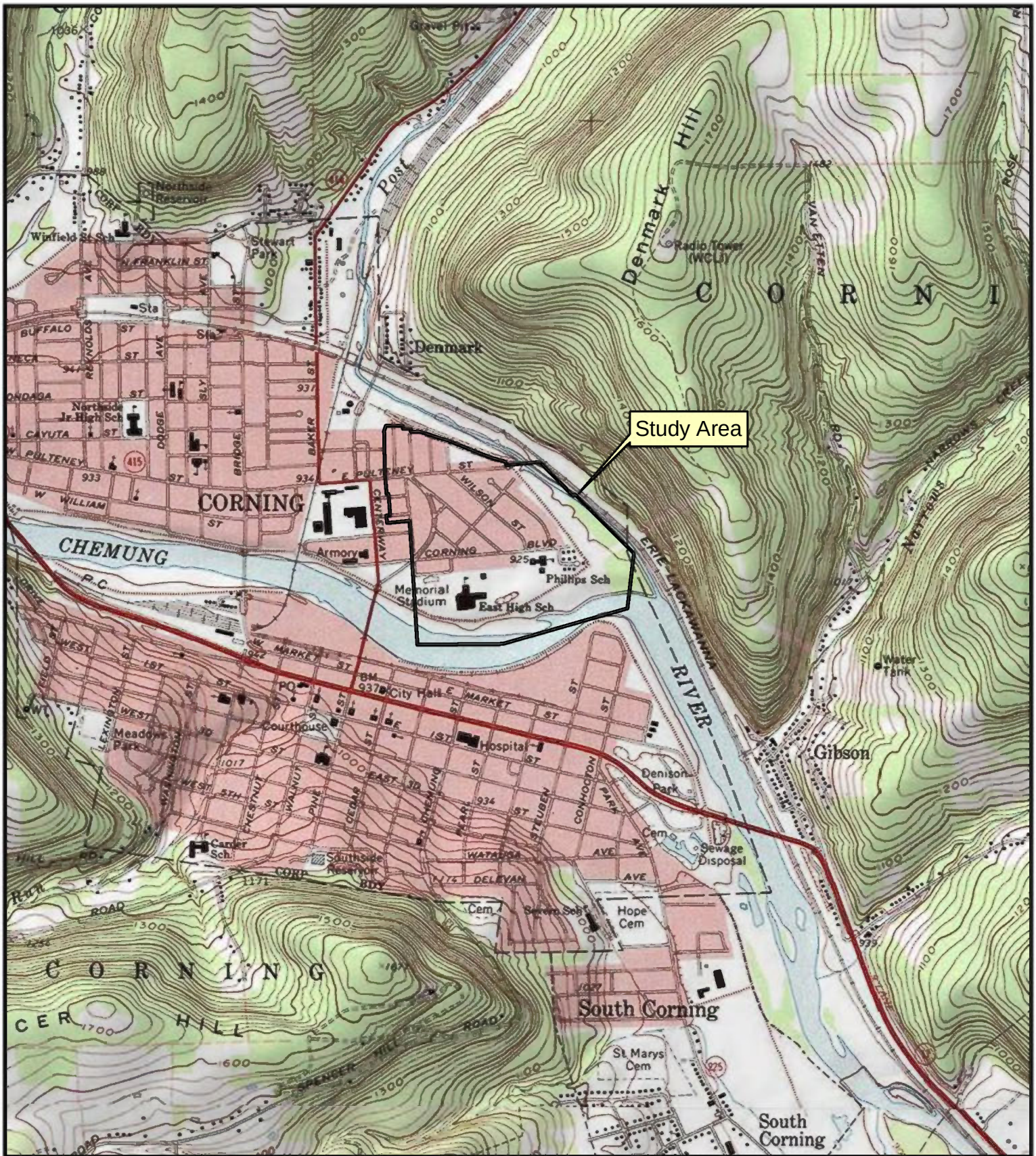
Tables and figures are provided at the end of each section for ease of review.

A Health and Safety Plan (HASP), Community Air Monitoring Plan (CAMP), Quality Assurance Project Plan (QAPP), and Standard Operating Procedures (SOPs) are included in the Appendices of this PDI Work Plan.



SECTION 1

FIGURES



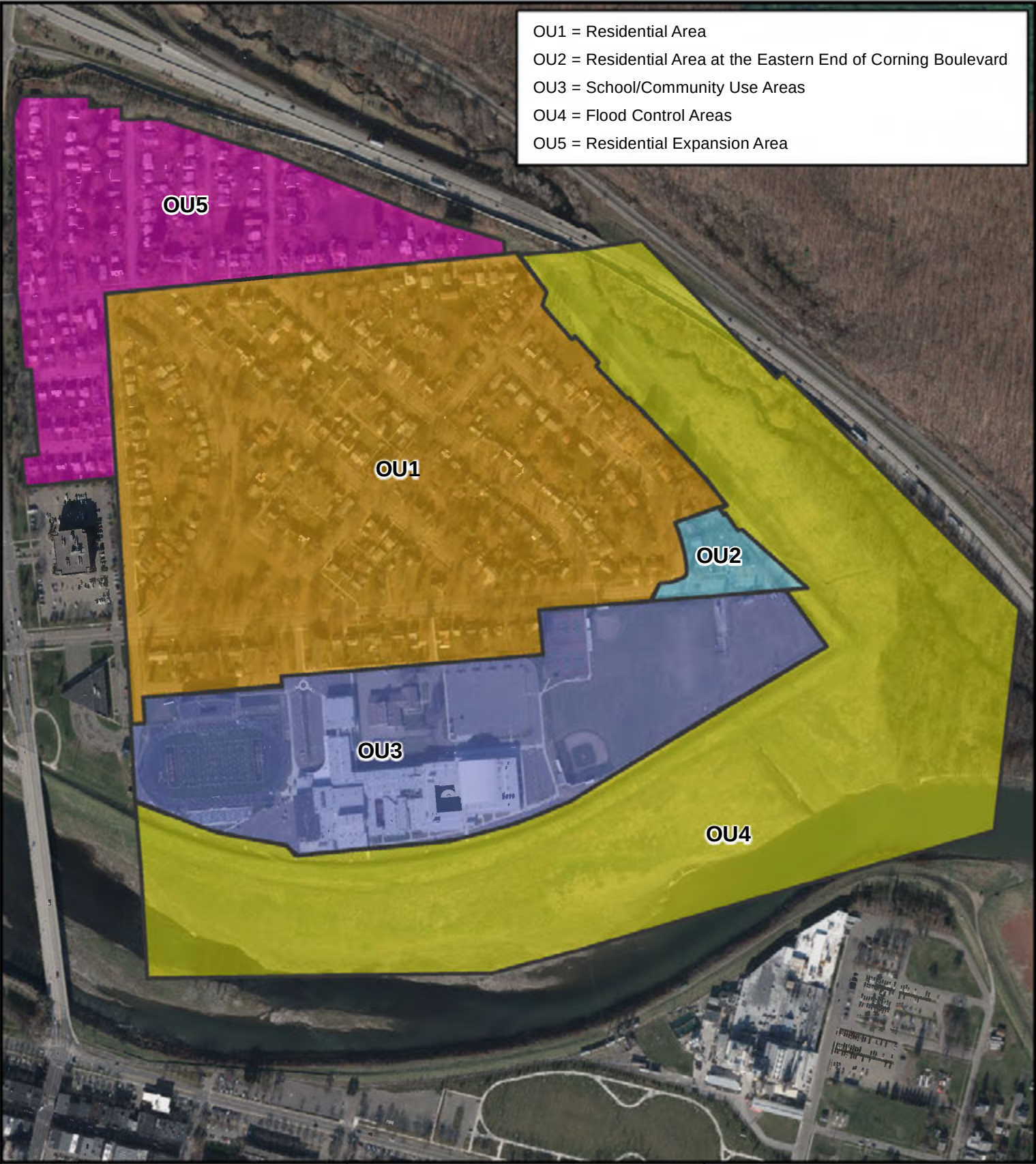
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WESTON
SOLUTIONS

0 1,000 2,000
Feet

USGS 7.5 Minute Quadrangle Corning NY, 1971

Figure 1-1
Location of Study Area
Corning, NY



OU1 = Residential Area
OU2 = Residential Area at the Eastern End of Corning Boulevard
OU3 = School/Community Use Areas
OU4 = Flood Control Areas
OU5 = Residential Expansion Area

Legend Operable Units - OU1 - OU2 - OU3 - OU4 - OU5	2 WESTON SOLUTIONS 0 250 500 Feet Base Imagery: Robinson Aerial Imagery, Dec 2015 Coordinate System: NAD 1983 State Plane New York Central Feet Datum: NAD83. Units: Feet	Figure 1-2 Study Area Operable Units Corning, NY



2. STUDY AREA DESCRIPTION AND HISTORY

2.1 STUDY AREA LOCATION AND DESCRIPTION

The Study Area is located in the City of Corning, New York, as illustrated on Figure 1-1. In general, it is bounded by the Chemung River to the south; Post Creek and Interstate 86 to the east and north; and the Guthrie Medical Center, the City of Corning Fire Department, and Centerway to the west. The Study Area is separated into five OUs, which are depicted in Figure 1-2:

- OU1 – Residential Area (includes 212 residential properties)
- OU2 – Residential Area at the Eastern End of Corning Boulevard (includes five residential properties)
- OU3 – School/Community Use Areas (includes the Corning-Painted Post School District, Corning Christian Academy and City of Corning Memorial Stadium properties)
- OU4 – Flood Control Areas
- OU5 – Residential Expansion Area (includes 109 residential properties)

2.2 STUDY AREA HISTORY

The City of Corning has a long history of manufacturing, particularly in brick and glassmaking. Historical references indicate that, in the late 1800s and early 1900s, one of the country's largest brick manufacturers and more than sixty glass manufacturers were located in the City of Corning (Dimitroff, 2001) (Sinclair & Spillman, 1997), including Corning Incorporated, which was formerly known as Corning Glass Works. During that time frame, coal was the primary fuel source in the Corning, New York area, and most of the local industries and municipalities used coal to heat their furnaces. In the early 1900s, when natural gas was introduced to the region, some industries converted their fuel sources to natural gas.

Between 1949 and at least 1968, the City of Corning operated a municipal incinerator that created significant volumes of ash. Historical City Council meeting minutes indicate that the City applied ash and cinders to roadways within the City to control ice during the winter months during, at least, the mid-1950s (City of Corning, 1936; 1941; 1958; 1959). These records also indicate that when land within the Study Area (now comprising OU3) was being considered for redevelopment as a



school in the late 1950s, the City of Corning stated that it would require “a considerable amount of work and expense involved in filling and grading to render the track suitable for recreational and educational purposes.” (City of Corning, 1950). Ultimately a school, which opened in 1962, was constructed on this portion of the Study Area.

Several times during the City of Corning’s history, the Chemung River overflowed its banks. This resulted in construction and improvement of flood control structures within the Study Area on multiple occasions, including in the mid-1940s and again after Hurricane Agnes in the mid-1970s, according to NYSDEC and U.S. Army Corps of Engineers records (USACE, 1941; USACE, 1973). Such construction efforts would have likely required the import of significant volumes of material of uncertain origin, the removal or relocation of material deemed unsuitable as foundation for earthworks, the creation and filling of borrow areas from which soils suitable for construction were obtained, as well as other potential grading and filling activities. These activities occurred within and surrounding the perimeters of the Study Area, including along the Chemung River, along Post Creek, and along what is now Interstate 86 (USACE, 1941; USACE, 1973).

The land use within OU1, OU2 and OU5 has developed over time from farmland into a residential area. In general, aerial photographs indicate that the development of OU1, OU2 and OU5 north of Corning Boulevard began prior to 1938 along Pyrex Street and Houghton Circle. The residential area subsequently expanded in an easterly direction across farmlands until about 1964, by which time OU1, OU2 and OU5 were mostly developed. During development activities, fill material was commonly used as sub-grade material for construction, to fill in low-lying areas and as an aid to drainage.

Through a title search of property deeds, it was found that part of the Study Area¹ encompasses lands previously owned by Corning Homes, Inc. (a residential developer not affiliated with Corning Incorporated). The deeds for these properties contained a condition that allowed Corning Glass Works (not a party to the transaction) to maintain structures, buildings and “ash dumps as now located” on the properties. Despite reviewing available historical maps, aerial photographs,

¹ This portion includes OU1, OU2, OU3 and OU4.



documents and public records, Corning Incorporated has not, to date, located any maps or records that depict the location, if any, of potential “ash dumps” as referenced in the deeds (i.e., that may have existed as of 1920).

In 2012, during a capital improvement project at the Corning-Painted Post High School located in OU3, within the southern portion of the Study Area, fill material that the Corning-Painted Post School District described as containing ash, brick, and glass, was encountered in the subsurface soils. During the capital improvement project, the Corning-Painted Post School District’s consultant tested excavated material to determine appropriate disposal methods. A review of a summary of the analytical results for these samples, prepared by the Corning-Painted Post School District’s consultant for NYSDEC, indicates over 200 samples were collected and analyzed for various constituents. The majority of the constituents were either not detected or were reported at concentrations below SCOs. The primary constituents which exceeded the SCOs in the excavated materials were lead, cadmium, and arsenic.

In the context of this PDI Work Plan, the term “fill” is used in several contexts. First, the term is used to refer to sub-grade construction material such as that found at the high school property and other material containing brick, ash, and/or glass (i.e., “fill material containing ash, brick and/or glass”). The term is also used to describe cover or backfill such as top soil and clay or sand that is brought in to support lawn and garden growth. The term “backfill” and/or “cover” will be used in this PDI Work Plan to describe materials that may be returned to an excavation or brought in as part of the Work to distinguish such materials from other references to previously existing fill material.

2.3 SUMMARY OF REMEDIAL INVESTIGATION

Commencing in June 2014, WESTON performed characterization activities within the Study Area, under the June 2014 Order on Consent. Characterization activities were performed in accordance with the Study Area Work Plan as modified, amended and approved by NYSDEC. Sampling activities were also performed by NYSDEC in OU1 and OU2 in 2014 and 2015. In 2015, Arcadis CE, Inc., on behalf of NYSDEC performed characterization activities in OU5.



The objectives of the Study Area characterization activities were to assess the nature and extent of layers of fill material containing ash, brick and/or glass that were encountered within the Study Area, and to collect data necessary for understanding the current conditions and associated potential exposure pathways. Validated analytical data for samples collected by WESTON in OU1 and OU2 of the Study Area have been submitted to NYSDEC in Data Usability Summary Reports (DUSRs) throughout the characterization program. NYSDEC has provided data to WESTON for its characterization activities in OU1, OU2 and OU5.

The Residential Areas of the Study Area consist of three OUs: OU1 – Residential Area (including 211 residential properties and Houghton Park), OU2 – Residential Area at the Eastern End of Corning Boulevard (including five residential properties) and OU5 – Residential Expansion Area (including 109 residential properties).

Characterization activities in the Residential Areas are substantially complete. A summary of findings of the investigations to date in OU1, OU2 and OU5 is described below:

- A total of 310 of the 326 properties have been sampled to date. This includes 201 of the 212 properties within OU1, the 5 properties within OU2 and 104 of 109 properties in OU5. Access to the remaining 16 properties has not been granted by property owners. Therefore these properties have not been sampled.
 - Laboratory analytical data for 171 of the 310 properties sampled indicated that soil concentrations are less than or equal to the SCOs. Of these 171 properties, the NYSDEC issued No Further Action letters for 33 properties in OU1 and 26 properties in OU5 on May 18, 2017. Additionally, NYSDEC issued No Further Action letters for 56 of these properties in OU1 on September 26, 2017.
- A total of 102 soil borings have been advanced in the City of Corning rights-of-way (ROWs) within OU1, OU2 and OU5.
 - Laboratory analytical data for samples collected from 64 of the 102 ROW soil borings indicated that soil concentrations are less than or equal to the SCOs.
- A total of 17 test pits were excavated in the Study Area
 - Laboratory analytical data for samples collected from 11 of the 17 test pits indicated that soil concentrations are less than or equal to the SCOs.



To date, more than 4,400 soil samples, including surface soil and soil boring samples, have been collected in OU1, OU2 and OU5, the majority of which had analytical results below the SCOs. These samples include surface soil and soil boring samples collected on individual residential properties and soil boring samples collected in the City of Corning ROWs. Laboratory analytical results for compounds detected at concentrations greater than the SCOs are summarized in Table 2-1, Table 2-2 and Table 2-3 for OU1, OU2 and OU5, respectively. For samples with analytical results above the SCOs, arsenic, cadmium, and lead (the constituents of potential concern or COPCs in the Study Area) were the constituents detected at concentrations greater than the SCOs most frequently.. Other constituents, as shown in Table 2-1 through Table 2-3, have also been detected in a limited number of samples at concentrations greater than SCOs.

Surface soil samples were collected from 0 to 2 inches below ground surface (in bgs). A total of 1,549 surface soil samples have been collected in OU1, OU2 and OU5: 1,137 in OU1; 1 in OU2; and 411 in OU5.

- Laboratory analytical data for 1,360 of the 1,549 surface soil samples were less than or equal to SCOs.
- Laboratory analytical data for 189 of the surface soil samples were greater than SCOs (139 surface soil samples in OU1, no surface soil samples in OU2 and 50 surface soil samples in OU5).

To date, a total of 1,011 soil borings were advanced in OU1, OU2 and OU5 to investigate sub-surface soil conditions. All soil borings were logged, and the presence of a layer of fill material containing ash, brick and/or glass was documented where encountered. The total number of soil borings advanced in OU1, OU2 and OU5 consist of:

- In OU1, 551 soil borings were advanced on residential properties and 71 soil borings were advanced in ROW areas (three of the soil borings advanced on residential properties, six of the soil borings advanced in the City of Corning rights-of-way (ROW) areas, and two of the soil borings advanced in a public park in OU1 were associated with test pit activities as discussed below).
- In OU2, 13 soil borings were advanced on residential properties and 2 soil borings were advanced in ROW areas.

- In OU5, 339 soil borings were advanced on residential properties and 35 soil borings were advanced in ROW areas.

The findings of the soil boring investigations in OU1, OU2 and OU5 related to the presence or absence of a layer of fill material containing ash, brick and/or glass are summarized in Table 2-4, Table 2-5, and Table 2-6, respectively. These tables categorize the soil borings by presence of a layer of fill material containing ash, brick and/or glass, as well as the depth at which analytical results were greater than SCOs.

A total of 678 of the 1,011 soil borings advanced in OU1, OU2 and OU5 to date contained no layer of fill material containing ash, brick and/or glass. Of the remaining 333 soil borings, a layer of fill material containing ash, brick and/or glass was encountered at the following depth intervals:

- From 0 to 2 ft bgs in 61 soil borings;
- From 0 to 2 ft bgs and below 2 ft bgs in 130 soil borings; and
- Greater than 2 ft bgs only in 142 soil borings.

A total of 17 test pits were excavated in the Study Area: five in OU1 ROWs, two in a public park located in OU1, three on residential properties within OU1, and seven in OU5 ROWs. A layer of fill material containing ash, brick and/or glass was encountered in one test pit excavated in a ROW in OU1 and in four of the test pits excavated in ROWs in OU5.

Garden soil samples, comprised of composite soil samples², were collected from 0 to 12 in bgs within vegetable gardens on residential properties in OU1 and OU5, and in non-vegetable gardens when requested by NYSDEC. No garden soil samples were collected in OU2. A total of 88 garden soil samples were collected, 73 of which contained concentrations less than or equal to SCOs. Of the 15 garden soil samples that had concentrations of one or more constituent greater than a SCO, 5 samples were collected in OU1 and 10 samples were collected in OU5.

Two rounds of groundwater samples were collected from seven groundwater monitoring wells installed in the Study Area and one irrigation groundwater well located in OU3. Arsenic, cadmium,

² Each composite garden soil sample consisted of 5 to 7 subsamples mixed together as one sample.



and lead, the constituents most frequently detected in the soils at concentrations greater than the SCOs in the Study Area, were not detected in groundwater at concentrations greater than New York State Division of Water Technical & Operational Guidance Series (TOGS) standards. This indicates that groundwater in the Study Area has not been impacted by layers of fill material containing ash, brick, and/or glass.



SECTION 2

TABLES



Table 2-1
Summary of Detected Analytical Results - OU1¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Residential Properties								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Barium, TCLP	100	0.16 J	105	415	5	410	1 ³	1
Cadmium, TCLP	1	0.00050 U	3.7	415	96	319	2 ³	2
Lead, TCLP	5	0.0030 U	277	415	126	289	31 ³	17
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	1.5 J	1280	3018	1	3017	255	57
Barium, Total	350	29.7	35600	2220	0	2220	34	21
Cadmium, Total	2.5	0.032 U	21000	3015	294	2721	127	40
Chromium, Total	36	3.0	556	2227	0	2227	22	12
Copper, Total	270	6.3	12900	2220	0	2220	4	4
Lead, Total	400	7.1	28200	3015	0	3015	112	40
Manganese, Total	2000	44.9	2720	2220	0	2220	3	3
Mercury, Total	0.81	0.0080 U	70.0 J	2188	69	2119	54	20
Nickel, Total	140	4.0 J	221	2220	0	2220	3	3
Selenium, Total	36	0.39 U	11200	2220	1370	850	4	4
Zinc, Total	2200	34.7	6260	2220	0	2220	3	3
<i>Semi-Volatile Organic Compounds (SVOCs), ug/Kg</i>								
2-Methylnaphthalene	410	2.3 U	2100 U	451	433	18	3	3
Benz(a)anthracene	1000	3.4 U	21000	451	268	183	48	24
Benzo(a)pyrene	1000	5.4 U	25000 J	451	272	179	50	26
Benzo(b)fluoranthene	1000	4.3 U	44000	451	263	188	58	28
Benzo(k)fluoranthene	1000	2.1 U	18000	451	319	132	31	18
Chrysene	1000	2.0 U	30000	451	311	140	49	25
Dibenz(a,h)anthracene	330	2.2 U	2500 J	451	416	35	5	5
Indeno(1,2,3-cd)pyrene	500	9.6 J	10000	451	265	186	77	29
Rights of Way Areas								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Lead, TCLP	5	0.0030 U	90.0	96	40	56	6 ³	2
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	0.50 U	429	177	1	176	34	2
Barium, Total	400	34.5	498	125	0	125	2	1



Table 2-1 (continued)
Summary of Detected Analytical Results - OU1¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Rights of Way Areas (continued)								
Total Metals, mg/Kg (continued)								
Cadmium, Total	4.3	0.031 U	48.0	177	13	164	14	2
Lead, Total	400	0.30 U	9480	177	1	176	16	2
Mercury, Total	0.81	0.0088 U	2.9	112	1	111	1	1
Semi-Volatile Organic Compounds (SVOCs), ug/Kg								
Benz(a)anthracene	1000	3.1 U	41000	97	45	52	9	2
Benzo(a)pyrene	1000	4.5 U	38000	97	38	59	10	2
Benzo(b)fluoranthene	1000	3.6 U	65000	97	34	63	12	2
Benzo(k)fluoranthene	3900	2.1 U	30000	97	45	52	2	1
Chrysene	3900	1.9 U	38000	97	43	54	2	1
Dibenz(a,h)anthracene	330	2.1 U	1800 U	97	65	32	3	1
Indeno(1,2,3-cd)pyrene	500	5.2 U	33000	97	40	57	9	2

Notes:

¹ Table includes compounds detected at concentrations greater than applicable screening levels in one or more sample.

² TCLP samples are screened against the TCLP Regulatory Screening Levels contained in the Code of Federal Regulations, Section 2.6.1-Toxicity Characteristic. Non-TCLP Samples from Residential Areas are screened against NYSDEC Subpart 375-6 Remedial Program Residential SCOs. Non-TCLP Samples from Rights-of-Way Areas are screened against NYSDEC Subpart 375-6 Remedial Program Restricted-Residential SCOs.

³ USEPA TCLP method is used to determine the appropriate disposal method under current NYSDEC and USEPA regulations.

⁴ Total Number of Samples above the Screening Level includes only detected concentrations above the screening level.

NYSDEC = New York State Department of Environmental Control

USEPA = United States Environmental Protection Agency

SCO = soil cleanup objective

TCLP = Toxicity Characteristic Leaching Procedure

OU = Operable Unit

mg/L = milligram per liter

µg/Kg = microgram per kilogram

mg/Kg = milligram per kilogram

U = The analyte was analyzed for, but was not detected above the level of the detection quantitation limit shown.

J = The positive result reported is estimated either because the result is less than the limit of quantitation (LOQ) or because certain quality control criteria were not met.



Table 2-2
Summary of Detected Analytical Results - OU2¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Residential Properties								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Cadmium, TCLP	1	0.00050 U	1.2	30	3	27	1 ³	1
Lead, TCLP	5	0.0030 U	35	30	5	25	5 ³	4
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	4.9	200	49	0	49	17	5
Barium, Total	350	71	660	30	0	30	1	1
Cadmium, Total	2.5	0.061 J	44	49	0	49	17	5
Chromium, Total	36	6.2	43	30	0	30	1	1
Lead, Total	400	9.3	4200	49	0	49	12	5
Nickel, Total	140	9.6	170	30	0	30	1	1
<i>Semi-Volatile Organic Compounds (SVOCs), ug/Kg</i>								
Benz(a)anthracene	1000	3.4 U	10000	37	11	19	5	4
Benzo(a)pyrene	1000	4.9 UJ	8700	37	7	23	5	4
Benzo(b)fluoranthene	1000	4.0 UJ	14000	37	6	24	5	4
Benzo(k)fluoranthene	1000	2.0 U	3800 J	37	14	16	3	2
Chrysene	1000	2.0 U	10000	37	8	22	5	4
Dibenz(a,h)anthracene	330	2.3 U	2500 J	37	12	18	4	3
Indeno(1,2,3-cd)pyrene	500	5.4 U	9900	37	10	20	5	4
Rights of Way Areas								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Lead, TCLP	5	0.010 U	8.7	4	2	2	1 ³	1
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	7.0	170	8	0	8	3	1
Cadmium, Total	4.3	0.16 J	53	8	0	8	3	1
Lead, Total	400	19	7600	8	0	8	3	1



Table 2-2 (continued)
Summary of Detected Analytical Results - OU2¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Rights of Way Areas (continued)								
Semi-Volatile Organic Compounds (SVOCs), ug/Kg								
Benz(a)anthracene	1000	3.5 U	3600	4	1	3	2	1
Benzo(a)pyrene	1000	4.9 U	7000	4	1	3	2	1
Benzo(b)fluoranthene	1000	6.5 J	8300	4	0	4	2	1
Chrysene	3900	2.1 U	4300	4	1	3	1	1
Dibenz(a,h)anthracene	330	2.4 U	2100	4	1	3	2	1
Indeno(1,2,3-cd)pyrene	500	5.7 U	8300	4	1	3	2	1

Notes:

¹ Table includes compounds detected at concentrations greater than applicable screening levels in one or more sample.

² TCLP samples are screened against the TCLP Regulatory Screening Levels contained in the Code of Federal Regulations, Section 2.6.1-Toxicity Characteristic. Context: Title 40- Protection of Environment. CHAPTER I - ENVIRONMENTAL PROTECTION AGENCY. SUBCHAPTER I- SOLID WASTES. PART 261 - IDENTIFICATION AND LISTING OF HAZARDOUS WASTE. Subpart G Characteristics of Hazardous Waste (2012-07-01). Non-TCLP Samples from Residential Areas are screened against NYSDEC Subpart 375-6 Remedial Program Residential SCOs. Non-TCLP Samples from Rights-of-Way Areas are screened against NYSDEC Subpart 375-6 Remedial Program Restricted-Residential SCOs.

³ USEPA TCLP method is used to determine the appropriate disposal method under current NYSDEC and USEPA regulations.

⁴ Total Number of Samples above the Screening Level includes only detected concentrations above the screening level.

NYSDEC = New York State Department of Environmental Control

USEPA = United States Environmental Protection Agency

SCO = soil cleanup objective

TCLP = Toxicity Characteristic Leaching Procedure

OU = Operable Unit

mg/L = milligram per liter

µg/Kg = microgram per kilogram

mg/Kg = milligram per kilogram

U = The analyte was analyzed for, but was not detected above the level of the detection quantitation limit shown.

J = The positive result reported is estimated either because the result is less than the limit of quantitation (LOQ) or because certain quality control criteria were not met.

UJ = The analyte was analyzed for, but not detected. The quantitation limit is approximate and may be inaccurate or imprecise.



Table 2-3
Summary of Detected Analytical Results - OU5¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Residential Properties								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Barium, TCLP	100	0.11 J	104	282	0	282	1 ³	1
Cadmium, TCLP	1	0.012 U	17.2	282	215	67	4 ³	4
Lead, TCLP	5	0.021 U	283	282	59	223	15 ³	10
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	0.89	669	1329	0	1329	138	53
Barium, Total	350	8.8 J	4790	1329	0	1329	35	19
Cadmium, Total	2.5	0.098 U	374	1329	1038	291	47	23
Chromium, Total	36	1.4 J	436	1329	1	1328	22	17
Copper, Total	270	2.4 J	738	1329	0	1329	5	4
Lead, Total	400	0.97	28600	1329	0	1329	71	31
Manganese, Total	2000	30.8	8260	1329	0	1329	10	10
Mercury, Total	0.81	0.011 U	18	1329	67	1262	31	21
Nickel, Total	140	1.5 J	442	1329	1	1328	2	2
Selenium, Total	36	0.35 U	65.8	1329	976	353	1	1
Zinc, Total	2200	10.1	14200	1329	0	1329	5	5
<i>Semi-Volatile Organic Compounds (SVOCs), ug/Kg</i>								
2-Methylnaphthalene	410	8.3 J	1900 U	282	127	155	7	6
Benz(a)anthracene	1000	32 J	5000	282	124	158	18	12
Benzo(a)pyrene	1000	12 J	5400	282	100	182	19	11
Benzo(b)fluoranthene	1000	15 J	11000 J	282	94	188	25	15
Benzo(k)fluoranthene	1000	17 J	3000	282	124	158	10	9
Chrysene	1000	11 J	8400 U	282	92	190	21	14
Dibenz(a,h)anthracene	330	21 J	1600	282	175	107	17	12
Indeno(1,2,3-cd)pyrene	500	24 J	7000	282	123	159	35	20
Isophorone	100000	8.4 J	150000	282	251	31	1	1
Rights of Way Areas								
<i>Toxicity Characteristic Leaching Procedure (TCLP) Metals, mg/L</i>								
Lead, TCLP	5	0.021 U	149	54	13	41	1 ³	1
<i>Total Metals, mg/Kg</i>								
Arsenic, Total	16	3.3 J	265	112	1	111	12	4
Cadmium, Total	4.3	0.33 U	54.1	112	99	13	2	2
Copper, Total	270	6.5	1520	112	1	111	1	1



Table 2-3 (continued)
Summary of Detected Analytical Results - OU5¹
Study Area, Corning, New York

	Screening Levels ²	Minimum Analytical Result	Maximum Analytical Result	Total # Samples	Total # Non-Detects	Total # Samples with Detected Concentrations	Total # Samples above the Screening Level ⁴	Total # Properties with Samples above the Screening Level
Rights of Way Areas (continued)								
Total Metals, mg/Kg (continued)								
Lead, Total	400	3.4	4030	112	0	112	5	2
Manganese, Total	2000	7.3	3390	112	0	112	3	2
Mercury, Total	0.81	0.012 U	1.3	112	9	103	1	1
Semi-Volatile Organic Compounds (SVOCs), ug/Kg								
Benzo(a)anthracene	1000	35 U	55000	54	24	30	4	2
Benzo(a)pyrene	1000	13 J	50000	54	21	33	4	2
Benzo(b)fluoranthene	1000	16 NJ	83000	54	19	35	7	4
Benzo(k)fluoranthene	3900	18 J	35000	54	24	30	1	1
Chrysene	3900	13 J	53000	54	16	38	2	2
Dibenz(a,h)anthracene	330	25 J	11000	54	27	27	3	2
Indeno(1,2,3-cd)pyrene	500	27 J	53000	54	24	30	11	4

Notes:

¹ Table includes compounds detected at concentrations greater than applicable screening levels in one or more sample.

² TCLP samples are screened against the TCLP Regulatory Screening Levels contained in the Code of Federal Regulations, Section 2.6.1-Toxicity Characteristic. Context: Title 40- Protection of Environment. CHAPTER I - ENVIRONMENTAL PROTECTION AGENCY. SUBCHAPTER I- SOLID WASTES. PART 261 - IDENTIFICATION AND LISTING OF HAZARDOUS WASTE. Subpart G Characteristics of Hazardous Waste (2012-07-01). Non-TCLP Samples from Residential Areas are screened against NYSDEC Subpart 375-6 Remedial Program Residential SCOs. Non-TCLP Samples from Rights-of-Way Areas are screened against NYSDEC Subpart 375-6 Remedial Program Restricted-Residential SCOs.

³ USEPA TCLP method is used to determine the appropriate disposal method under current NYSDEC and USEPA regulations.

⁴ Total Number of Samples above the Screening Level includes only detected concentrations above the screening level.

NYSDEC = New York State Department of Environmental Control

USEPA = United States Environmental Protection Agency

SCO = soil cleanup objective

TCLP = Toxicity Characteristic Leaching Procedure

OU = Operable Unit

mg/L = milligram per liter

µg/Kg = microgram per kilogram

mg/Kg = milligram per kilogram

U = The analyte was analyzed for, but was not detected above the level of the detection quantitation limit shown.

J = The positive result reported is estimated either because the result is less than the limit of quantitation (LOQ) or because certain quality control criteria were not met.

NJ = The detection is tentative in identification and estimated value. Although there is presumptive evidence of the analyte, the result should be used with caution as a potential false positive and/or elevated quantitative value.



Table 2-4
Soil Boring Summary - OU1¹
Study Area, Corning, NY

Sample Results	Observations			
	No Layer of fill material containing ash, brick and/or glass observed	Layer of fill material containing ash, brick and/or glass observed		
		0-2 ft bgs Only	0-2 ft bgs and > 2 ft bgs	> 2 ft bgs Only
Number of Soil Borings with All Soil Samples < SCOs ²	378	10	6	18
Number of Soil Borings with One or More Soil Samples >SCOs in 0-2 ft bgs Only ²	36	18	2	7
Number of Soil Borings with One or More Soil Sample >SCOs in 0-2 ft bgs and > 2 ft bgs ²	5	2	27	34
Number of Soil Borings with One or More Soil Sample >SCOs in >2 ft bgs Only ²	5	2	10	53
Number of Soil Borings with no Samples Collected ³	9	0	0	0
Totals	433	32	45	112
		189		

Notes:

¹ Includes 551 soil borings on residential properties, 71 soil borings in the City of Corning ROW areas

² Samples collected in residential soil borings compared to residential SCOs and samples collected in ROW area soil borings compared to restricted residential SCOs.

³ Soil borings were associated with test pit activities and because no layer of ash, brick and/or glass was observed in these soil borings, no samples were collected.

ft bgs = feet below ground surface

SCOs = New York State Department of Environmental Conservation (NYSDEC) soil cleanup objectives

ROW = Rights-of-Way



Table 2-5
Soil Boring Summary - OU2¹
Study Area, Corning, NY

Sample Results	Observations			
	No Layer of fill material containing ash, brick and/or glass observed	Layer of fill material containing ash, brick and/or glass observed		
		0-2 ft bgs Only	0-2 ft bgs and > 2 ft bgs	> 2 ft bgs Only
Number of Soil Borings with All Soil Samples < SCOs ²	4	0	0	1
Number of Soil Borings with One or More Soil Samples >SCOs in 0-2 ft bgs Only ²	0	0	0	0
Number of Soil Borings with One or More Soil Sample >SCOs in 0-2 ft bgs and > 2 ft bgs ²	0	0	0	4
Number of Soil Borings with One or More Soil Sample >SCOs in >2 ft bgs Only ²	0	0	0	6
Totals	4	0	0	11
		11		

Notes:

¹ Includes 13 soil borings on residential properties and 2 soil borings in the City of Corning ROW

² Samples collected in residential soil borings compared to residential SCOs and samples collected in ROW area soil borings compared to restricted residential SCOs.

ft bgs = feet below ground surface

SCOs = New York State Department of Environmental Conservation (NYSDEC) soil cleanup objectives

ROW = Rights-of-Way



Table 2-6
Soil Boring Summary - OU5¹
Study Area, Corning, NY

Sample Results	Observations			
	No Layer of fill material containing ash, brick and/or glass observed	Layer of fill material containing ash, brick and/or glass observed		
		0-2 ft bgs Only	0-2 ft bgs and > 2 ft bgs	> 2 ft bgs Only
Number of Soil Borings with All Soil Samples < SCOs ²	212	17	14	5
Number of Soil Borings with One or More Soil Samples >SCOs in 0-2 ft bgs Only ²	10	12	7	1
Number of Soil Borings with One or More Soil Sample >SCOs in 0-2 ft bgs and > 2 ft bgs ²	17	0	30	11
Number of Soil Borings with One or More Soil Sample >SCOs in >2 ft bgs Only ²	2	0	34	2
Totals	241	29	85	19
		133		

Notes:

¹ Includes 339 soil borings on residential properties and 35 soil borings in the City of Corning ROW

² Samples collected in residential soil borings compared to residential SCOs and samples collected in ROW area soil borings compared to restricted residential SCOs.

ft bgs = feet below ground surface

SCOs = New York State Department of Environmental Conservation (NYSDEC) soil cleanup objectives

ROW = Rights-of-Way



3. ENVIRONMENTAL SETTING

3.1 LAND USE

The Study Area consists of approximately 201 acres of land located on the eastern side of the City of Corning, New York along the northern bank of the Chemung River, northwest of the confluence with Post Creek (see Figure 1-1). The Study Area includes the Corning-Painted Post High School property; the Corning Christian Academy property; the City of Corning Memorial Stadium property; a residential area consisting of 326 individual properties; and flood control areas along the Chemung River and Post Creek. The properties within the Study Area are zoned as either public-conservation (PC) or low-density residential (R1) by the City of Corning as illustrated on Figure 3-1. The land area zoned PC is generally concentrated south of Corning Boulevard and in the flood control areas near the Chemung River and Post Creek. No industrial/commercial facilities are known to be located within the Study Area at present.

The 326 individual properties in OU1, OU2 and OU5 are zoned R1. Of these properties, 305 are classified as single family residences; one is classified as a single family residence with an apartment; seven are classified as multi-family residences; three are classified as apartments; nine are classified as residential-vacant land; and one (Houghton Park) is classified as a playground. A list of the individual properties that comprise OU1, OU2 and OU5, with the zoning, property classifications, and dates of construction is presented in Table 3-1. Zoning and property classifications are also illustrated on Figure 3-1 and Figure 3-2, respectively.

3.2 TOPOGRAPHY AND DRAINAGE

The Study Area is relatively flat with a slight gradient to the south and east. The Corning, New York 1976 U.S. Geological Service (USGS) 7.5-minute topographic quadrangle map indicates that the Study Area is approximately 929 feet above mean sea level (ft amsl). Within a one mile radius of the Study Area, the ground surface elevation ranges from 915 ft amsl to 1,459 ft amsl, with two steep elevation changes, one located to the north and one to the east.

Surface water within the Study Area generally flows south/southeast toward the Chemung River. Storm water is believed to be conveyed to the river through one or more storm drains located in the southeast corner of the Study Area. While the flow of Post Creek and the Chemung River have

changed over time and have been altered for flood control, drainage and development purposes, surface flow in these waterways has generally been south/southeast. Surface water from the confluence of Post Creek and the Chemung River flows southward to where it ultimately joins the Susquehanna River. Due to the proximity of the Chemung River and Post Creek, OU4 is located within both the Federal Emergency Management Agency (FEMA) 100-year and 500-year flood zones (FEMA, 2002).

The Chemung River flows along the southern portion of the Study Area and has a drainage area of approximately 2,006 square miles. Measured daily flows range from a minimum of 640 cubic feet per second (cfs) to 20,200 cfs, with median and mean flows of 1,820 and 3,620 cfs, based on 38 years of records. The Chemung River is designated as Class C water in the New York State classification system (USGS, 2014). Class C waters indicate the water is suitable for non-contact activities, and is best used for supporting fisheries.

The much smaller, second order Post Creek along the eastern edge of the Study Area also has a Class C designation in the vicinity of the Study Area. The riparian zone immediately adjacent to Post Creek is wooded.

3.3 GEOLOGY

The Study Area is located in the Chemung River valley, and contains predominately sand and gravel deposits of glaciofluvial origin and more recent alluvial deposits. The river valley deposits are on the order of 100 feet thick in the vicinity of the Study Area. These river valley deposits are underlain by low permeability shale/siltstone bedrock (Miller, 1982). In the vicinity of the Study Area, a low permeability, lacustrine silt and clay layer (approximately 10 feet thick) is present about 30 ft bgs (Miller, 1982).

3.4 HYDROGEOLOGY

The saturated portions of the Chemung River valley deposits are recharged principally by infiltration of precipitation. This valley-filled glacial/alluvial aquifer is generally unconfined (i.e., the water table forms the upper boundary of the aquifer) and saturated approximately to the level of nearby rivers (such as the Chemung River) (Olcot, 1995). In the higher topographic portions of the Study Area, the depth to the water table is expected to be on the order of 20 to 25 ft bgs;



however, groundwater levels may be deeper where supply wells actively extract groundwater from the valley aquifer. Groundwater in the valley aquifer generally flows toward and discharges to nearby rivers/creeks; however, groundwater flow directions can be locally altered by supply well withdrawals from the valley aquifer.

3.5 ECOLOGICAL SETTING

Much of the Study Area is composed of a terrestrial cultural ecological community created and maintained by human activities. It has been modified by human influence to such a degree that the physical conformation of the substrate and the biological composition of the resident ecological community is substantially different from the character of the substrate or community as it existed prior to human influence. The ground cover in OU1, OU2 and OU5 is primarily mowed lawn with trees.



SECTION 3

FIGURES



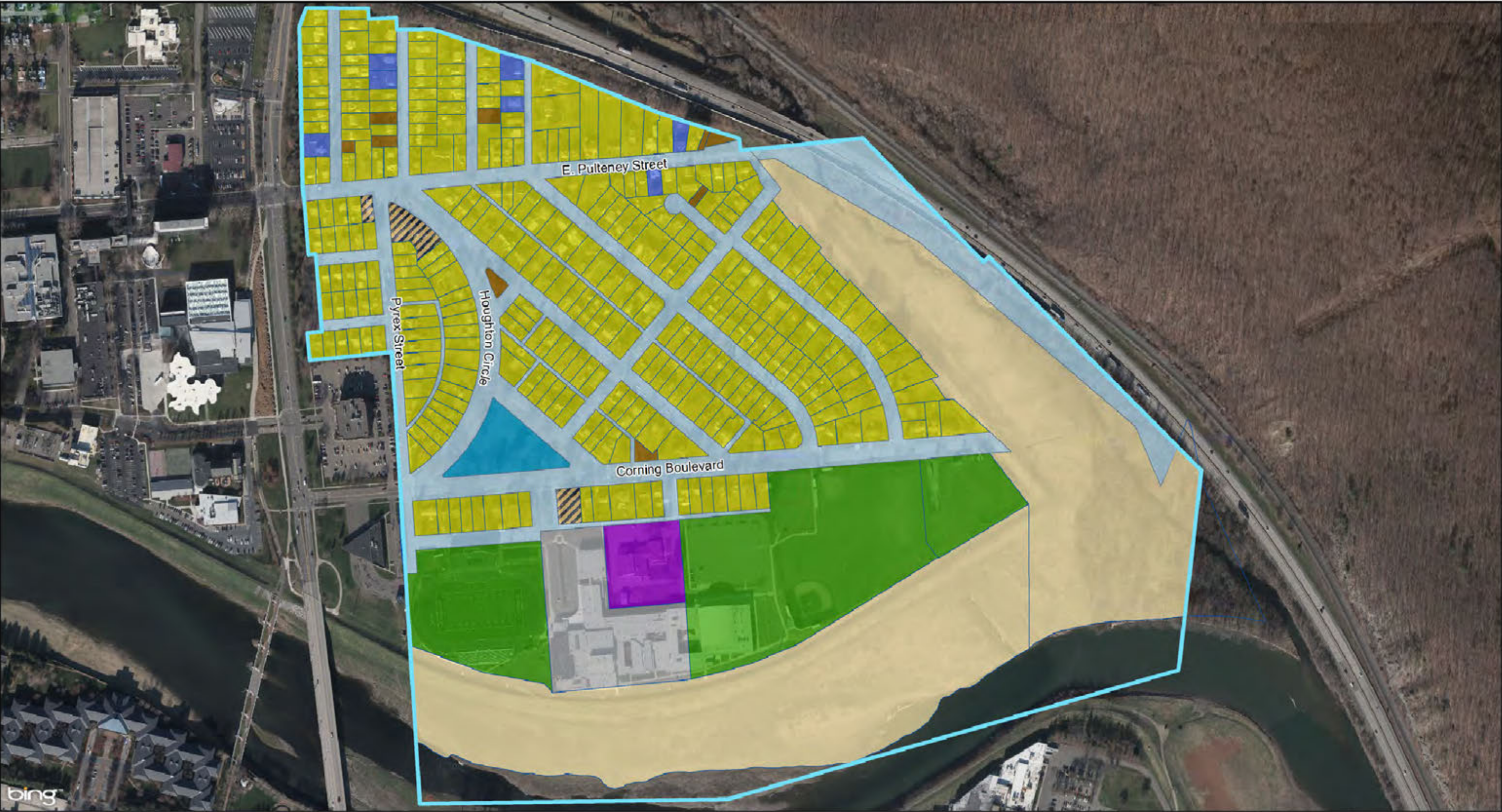
Manganese Results	
Legend	
Study Area	
Zoning	
Public-Conservation	
R1 - Low Density	

NOTES: Base Imagery: Robinson Aerial Imagery, Dec 2015 Coordinate System: NAD 1983 State Plane New York Central Feet Datum: NAD83. Units: Feet	
0 400 800 Feet	0 80 160 240 Meters

Study Area Corning, NY



Figure 3-1 Property Zoning	
Document Name: Zoning.MXD	
3/14/2017	



Legend

Study Area	591 - Playground	Road/City of Corning Rights-of-Way
Property Classification	592 - Athletic Field	
210 - Single Family Residence	612 - School	
220 - Multi Family Residence	615 - Educational Facility	
311 - Residential - Vacant Land	622 - Fire/Police	
411 - Apartment	821 - Flood Control	

Manganese Results

591 - Playground
592 - Athletic Field
612 - School
615 - Educational Facility
622 - Fire/Police
821 - Flood Control

NOTES:
Base Imagery: Robinson Aerial Imagery, Dec 2015
Coordinate System: NAD 1983 State Plane
New York Central Feet
Datum: NAD83. Units: Feet

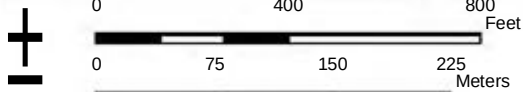
Study Area
Corning, NY



Figure 3-2
Property Classification

Document Name: Property_Classification.MXD

3/14/2017





SECTION 3

TABLE



Table 3-1
Property Information for OU1, OU2 and OU5
Study Area, Corning, New York

OU	Parcel ID	Address	Property Classification ¹	Year Built
OU1	318.05-01-001.000	Pershing St (Memorial Stone)	Residential - Vacant Land	---
OU1	318.05-01-002.000	69 Pershing St	Single Family Residence	1942
OU1	318.05-01-003.000	6 Belleau St	Single Family Residence	1928
OU1	318.05-01-004.000	4 Belleau St	Single Family Residence	1960
OU1	318.05-01-005.000	65 Pershing St	Single Family Residence	1926
OU1	318.05-01-006.000	61 Pershing St	Single Family Residence	1956
OU1	318.05-01-007.000	57 Pershing St	Single Family Residence	1928
OU1	318.05-01-008.000	53 Pershing St	Single Family Residence	1972
OU1	318.05-01-009.000	49 Pershing St	Single Family Residence	1926
OU1	318.05-01-010.000	47 Pershing St	Single Family Residence	1950
OU1	318.05-01-011.000	45 Pershing St	Single Family Residence	1973
OU1	318.05-01-012.000	35 Pershing St	Single Family Residence	1946
OU1	318.05-01-013.000	33 Pershing St	Single Family Residence	1927
OU1	318.05-01-014.000	29 Pershing St	Single Family Residence	1955
OU1	318.05-01-015.000	25 Pershing St	Single Family Residence	1926
OU1	318.05-01-016.000	21 Pershing St	Single Family Residence	1930
OU1	318.05-01-017.000	75 Corning Blvd	Single Family Residence	1940
OU1	318.05-01-018.000	29 Pershing St	Residential - Vacant Land	---
OU1	318.05-01-019.000	65 Corning Blvd	Single Family Residence	1939
OU1	318.05-01-020.000	33 Sims Ave	Single Family Residence	1999
OU1	318.05-01-021.000	31 Sims Ave	Single Family Residence	1943
OU1	318.05-01-022.000	27 Sims Ave	Single Family Residence	1930
OU1	318.05-01-023.000	21 Sims Ave	Single Family Residence	1922
OU1	318.05-01-024.000	19 Sims Ave	Single Family Residence	1943
OU1	318.05-01-025.000	17 Sims Ave	Single Family Residence	1940
OU1	318.05-01-026.000	15 Sims Ave	Single Family Residence	1956
OU1	318.05-01-027.000	11 Sims Ave	Single Family Residence	1940
OU1	318.05-01-028.000	42 Houghton Cir	Single Family Residence	1940
OU1	318.05-01-029.000	40 Houghton Cir	Single Family Residence	1910
OU1	318.05-01-031.000	Houghton Park	Playground	---
OU1	318.05-01-032.000	6 Corning Blvd	Single Family Residence	1973
OU1	318.05-01-033.000	10 Corning Blvd	Single Family Residence	1919
OU1	318.05-01-034.000	14 Corning Blvd	Single Family Residence	1920
OU1	318.05-01-035.000	18 Corning Blvd	Single Family Residence	1930
OU1	318.05-01-036.000	20 Corning Blvd	Single Family Residence	1940
OU1	318.05-01-037.000	22 Corning Blvd	Single Family Residence	1950
OU1	318.05-01-039.000	26 Corning Blvd	Single Family Residence	1974
OU1	318.05-01-040.000	40 Corning Blvd	Single Family Residence	1950
OU1	318.05-01-041.000	50 Corning Blvd	Apartment	---
OU1	318.05-01-042.000	54 Corning Blvd	Single Family Residence	1954
OU1	318.05-01-043.000	58 Corning Blvd	Single Family Residence	1940
OU1	318.05-01-044.000	66 Corning Blvd	Single Family Residence	1935
OU1	318.05-01-045.000	74 Corning Blvd	Single Family Residence	1928
OU1	318.05-01-046.000	78 Corning Blvd	Single Family Residence	1942
OU1	318.05-01-047.000	82 Corning Blvd	Single Family Residence	1930
OU1	318.05-01-048.000	84 Corning Blvd	Single Family Residence	1929
OU1	318.05-01-049.000	94 Corning Blvd	Single Family Residence	1952
OU1	318.05-01-050.000	100 Corning Blvd	Single Family Residence	1945
OU1	318.05-01-051.000	102 Corning Blvd	Single Family Residence	1937
OU1	318.05-01-052.000	104 Corning Blvd	Single Family Residence	1938
OU1	318.05-01-053.000	106 Corning Blvd	Single Family Residence	1947
OU1	318.05-02-046.000	67-99 Houghton Cir	Apartment	---
OU1	318.05-02-048.000	63 Houghton Cir	Single Family Residence	1950
OU1	318.05-02-049.000	61 Houghton Cir	Single Family Residence	1955
OU1	318.05-02-050.000	59 Houghton Cir	Single Family Residence	1952
OU1	318.05-02-051.000	55 Houghton Cir	Single Family Residence	1951
OU1	318.05-02-052.000	53 Houghton Cir	Single Family Residence	1920
OU1	318.05-02-053.000	49 Houghton Cir	Single Family Residence	1951
OU1	318.05-02-054.000	47 Houghton Cir	Single Family Residence	1952
OU1	318.05-02-055.000	45 Houghton Cir	Single Family Residence	1951
OU1	318.05-02-056.000	39 Houghton Cir	Single Family Residence	1928
OU1	318.05-02-057.000	33 Houghton Cir	Single Family Residence	1973
OU1	318.05-02-058.000	27 Houghton Cir	Single Family Residence	1930
OU1	318.05-02-059.000	21 Houghton Cir	Single Family Residence	1972
OU1	318.05-02-060.000	19 Houghton Cir	Single Family Residence	1937
OU1	318.05-02-061.000	15 Houghton Cir	Single Family Residence	1930
OU1	318.05-02-062.000	13 Houghton Cir	Single Family Residence	1945
OU1	318.05-02-063.000	11 Houghton Cir	Single Family Residence	1951



Table 3-1
Property Information for OU1, OU2 and OU5
Study Area, Corning, New York
(continued)

OU	Parcel ID	Address	Property Classification ¹	Year Built
OU1	318.05-02-064.000	9 Houghton Cir	Single Family Residence	1935
OU1	318.05-02-066.000	24 Pyrex St	Single Family Residence	1956
OU1	318.05-02-067.000	26 Pyrex St	Single Family Residence	1972
OU1	318.05-02-068.000	28 Pyrex St	Single Family Residence	1953
OU1	318.05-02-069.000	30 Pyrex St	Single Family Residence	1929
OU1	318.05-02-070.000	32 Pyrex St	Single Family Residence	1950
OU1	318.05-02-071.000	34 Pyrex St	Single Family Residence	1950
OU1	318.05-02-072.000	36 Pyrex St	Single Family Residence	1950
OU1	318.05-02-073.000	38 Pyrex St	Single Family Residence	1952
OU1	318.05-02-074.000	40 Pyrex St	Single Family Residence	1920
OU1	318.05-02-075.000	42 Pyrex St	Single Family Residence	1952
OU1	318.05-02-076.000	44 Pyrex St	Single Family Residence	1973
OU1	318.05-02-077.000	46 Pyrex St	Single Family Residence	1952
OU1	318.05-02-078.000	48 Pyrex St	Single Family Residence	1953
OU1	318.05-03-001.000	94 Pershing St	Single Family Residence	1946
OU1	318.05-03-002.000	92 Pershing St	Single Family Residence	1950
OU1	318.05-03-003.000	88 Pershing St	Single Family Residence	1950
OU1	318.05-03-004.000	86 Pershing St	Single Family Residence	1950
OU1	318.05-03-005.000	84 Pershing St	Single Family Residence	1974
OU1	318.05-03-006.000	82 Pershing St	Single Family Residence	1973
OU1	318.05-03-007.000	80 Pershing St	Single Family Residence	1973
OU1	318.05-03-008.000	66 Pershing St	Single Family Residence	1938
OU1	318.05-03-009.000	64 Pershing St	Single Family Residence	1973
OU1	318.05-03-010.000	62 Pershing St	Single Family Residence	1948
OU1	318.05-03-011.000	60 Pershing St	Single Family Residence	1943
OU1	318.05-03-012.000	58 Pershing St	Single Family Residence	1940
OU1	318.05-03-013.000	54 Pershing St	Single Family Residence	1945
OU1	318.05-03-014.000	50 Pershing St	Single Family Residence	1975
OU1	318.05-03-015.000	42 Pershing St	Single Family Residence	1925
OU1	318.05-03-016.000	99 Argonne St	Single Family Residence	1973
OU1	318.05-03-018.000	45 Wilson St	Single Family Residence	1975
OU1	318.05-03-019.000	47 Wilson St	Single Family Residence	1975
OU1	318.05-03-020.000	49 Wilson St	Single Family Residence	1948
OU1	318.05-03-022.000	53 Wilson St	Single Family Residence	1973
OU1	318.05-03-023.000	55 Wilson St	Single Family Residence	1973
OU1	318.05-03-025.000	59 Wilson St	Single Family Residence	1974
OU1	318.05-03-026.000	61 Wilson St	Single Family Residence	1974
OU1	318.05-03-028.000	65 Wilson St	Single Family Residence	1974
OU1	318.05-03-029.000	67 Wilson St	Single Family Residence	1952
OU1	318.05-03-030.000	232 E Pulteney St	Single Family Residence	1950
OU1	318.05-03-031.000	234 E Pulteney St	Single Family Residence	1945
OU1	318.05-03-032.000	236 E Pulteney St	Single Family Residence	1950
OU1	318.05-03-033.000	240 E Pulteney St	Single Family Residence	1973
OU1	318.05-03-034.000	244 E Pulteney St	Single Family Residence	1952
OU1	318.05-03-035.000	56 Wilson St	Single Family Residence	1973
OU1	318.05-03-036.000	54 Wilson St	Single Family Residence	1973
OU1	318.05-03-037.000	52 Wilson St	Single Family Residence	1973
OU1	318.05-03-038.000	50 Wilson St	Single Family Residence	1974
OU1	318.05-03-040.000	46 Wilson St	Single Family Residence	2013
OU1	318.05-03-041.000	44 Wilson St	Single Family Residence	1951
OU1	318.05-03-042.000	42 Wilson St	Single Family Residence	1950
OU1	318.05-03-043.000	34 Wilson St	Single Family Residence	1950
OU1	318.05-03-044.000	109 Argonne St	Single Family Residence	1977
OU1	318.05-03-045.000	111 Argonne St	Single Family Residence	1957
OU1	318.05-03-046.000	6 Jackson Cir	Single Family Residence	1940
OU1	318.05-03-047.000	8 Jackson Cir	Single Family Residence	1956
OU1	318.05-03-048.000	10 Jackson Cir	Single Family Residence	1945
OU1	318.05-03-049.000	12 Jackson Cir	Single Family Residence	1948
OU1	318.05-03-050.000	248 E Pulteney St	Single Family Residence	1952
OU1	318.05-03-051.000	250 E Pulteney St	Single Family Residence	1955
OU1	318.05-03-052.000	252 E Pulteney St	Single Family Residence	1973
OU1	318.05-03-053.000	258 E Pulteney St	Multi Family Residence	1927
OU1	318.05-03-054.000	262 E Pulteney St	Single Family Residence	1958
OU1	318.05-03-055.000	260 E Pulteney St	Single Family Residence	1927
OU1	318.05-03-056.000	264 E Pulteney St	Single Family Residence	1960
OU1	318.05-03-057.000	266 E Pulteney St	Single Family Residence	1960
OU1	318.05-03-058.000	268 E Pulteney St	Single Family Residence	1960
OU1	318.05-03-059.000	270 E Pulteney St	Single Family Residence	1959



Table 3-1
Property Information for OU1, OU2 and OU5
Study Area, Corning, New York
(continued)

OU	Parcel ID	Address	Property Classification ¹	Year Built
OU1	318.05-03-060.000	121 Argonne St	Single Family Residence	1973
OU1	318.05-03-062.000	117 Argonne St	Single Family Residence	1974
OU1	318.05-03-063.000	115 Argonne St	Single Family Residence	1956
OU1	318.05-03-064.000	113 Argonne St	Single Family Residence	1960
OU1	318.05-03-065.000	7 Jackson Cir	Single Family Residence	1946
OU1	318.05-03-066.000	17 Jackson Cir	Residential - Vacant Land	---
OU1	318.05-04-001.000	36 Pershing St	Single Family Residence	1937
OU1	318.05-04-002.000	34 Pershing St	Single Family Residence	1940
OU1	318.05-04-003.000	30 Pershing St	Single Family Residence	1925
OU1	318.05-04-004.000	26 Pershing St	Single Family Residence	1925
OU1	318.05-04-005.000	22 Pershing St	Single Family Residence	1927
OU1	318.05-04-006.000	18 Pershing St	Single Family Residence	1928
OU1	318.05-04-007.000	14 Pershing St	Single Family Residence	1937
OU1	318.05-04-008.000	10 Pershing St	Single Family Residence	1925
OU1	318.05-04-009.000	8 Pershing St	Single Family Residence	1969
OU1	318.05-04-010.000	105 Corning Blvd	Single Family Residence	1973
OU1	318.05-04-011.000	107 Corning Blvd	Single Family Residence	1947
OU1	318.05-04-012.000	109 Corning Blvd	Single Family Residence	1946
OU1	318.05-04-013.000	7 Wilson St	Single Family Residence	1948
OU1	318.05-04-014.000	9 Wilson St	Single Family Residence	1947
OU1	318.05-04-015.000	11 Wilson St	Single Family Residence	1948
OU1	318.05-04-016.000	13 Wilson St	Single Family Residence	1947
OU1	318.05-04-017.000	15 Wilson St	Single Family Residence	1947
OU1	318.05-04-018.000	17 Wilson St	Single Family Residence	1973
OU1	318.05-04-019.000	19 Wilson St	Single Family Residence	1947
OU1	318.05-04-020.000	21 Wilson St	Single Family Residence	1947
OU1	318.05-04-021.000	23 Wilson St	Single Family Residence	1949
OU1	318.05-04-022.000	25 Wilson St	Single Family Residence	1947
OU1	318.05-04-023.000	27 Wilson St	Single Family Residence	1947
OU1	318.05-04-024.000	29 Wilson St	Single Family Residence	1948
OU1	318.05-04-025.000	98 Argonne St	Single Family Residence	1973
OU1	318.05-04-026.000	104 Argonne St	Single Family Residence	1978
OU1	318.05-04-028.000	30 Wilson St	Single Family Residence	1947
OU1	318.05-04-029.000	28 Wilson St	Single Family Residence	1947
OU1	318.05-04-030.000	26 Wilson St	Single Family Residence	1948
OU1	318.05-04-031.000	24 Wilson St	Single Family Residence	1948
OU1	318.05-04-032.000	22 Wilson St	Single Family Residence	1951
OU1	318.05-04-033.000	20 Wilson St	Single Family Residence	1947
OU1	318.05-04-034.000	18 Wilson St	Single Family Residence	1947
OU1	318.05-04-035.000	16 Wilson St	Single Family Residence	1947
OU1	318.05-04-036.000	14 Wilson St	Single Family Residence	1946
OU1	318.05-04-037.000	12 Wilson St	Single Family Residence	1947
OU1	318.05-04-038.000	10 Wilson St	Single Family Residence	1942
OU1	318.05-04-039.000	8 Wilson St	Single Family Residence	1977
OU1	318.05-04-040.000	6 Wilson St	Single Family Residence	1947
OU1	318.05-04-041.000	4 Wilson St	Single Family Residence	1976
OU1	318.05-04-042.000	115 Corning Blvd	Single Family Residence	1950
OU1	318.05-04-043.000	117 Corning Blvd	Single Family Residence	1975
OU1	318.05-04-045.000	121 Corning Blvd	Single Family Residence	1975
OU1	318.05-04-046.000	7 Roosevelt St	Single Family Residence	1974
OU1	318.05-04-048.000	11 Roosevelt St	Single Family Residence	1974
OU1	318.05-04-049.000	15 Roosevelt St	Single Family Residence	1976
OU1	318.05-04-050.000	17 Roosevelt St	Single Family Residence	1977
OU1	318.05-04-051.000	19 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-052.000	21 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-054.000	25 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-055.000	27 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-056.000	33 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-057.000	35 Roosevelt St	Single Family Residence	1975
OU1	318.05-04-058.000	37 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-060.000	39 Roosevelt St	Single Family Residence	1975
OU1	318.05-04-061.000	108 Argonne St	Single Family Residence	1977
OU1	318.05-04-062.000	46 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-063.000	44 Roosevelt St	Single Family Residence	1951
OU1	318.05-04-064.000	42 Roosevelt St	Single Family Residence	1976
OU1	318.05-04-066.000	38 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-067.000	36 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-068.000	34 Roosevelt St	Single Family Residence	1973



Table 3-1
Property Information for OU1, OU2 and OU5
Study Area, Corning, New York
(continued)

OU	Parcel ID	Address	Property Classification ¹	Year Built
OU1	318.05-04-069.000	30 Roosevelt St	Single Family Residence	1978
OU1	318.05-04-070.000	28 Roosevelt St	Single Family Residence	1974
OU1	318.05-04-072.000	24 Roosevelt St	Single Family Residence	1976
OU1	318.05-04-073.000	20 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-074.000	18 Roosevelt St	Single Family Residence	1976
OU1	318.05-04-076.000	16 Roosevelt St	Single Family Residence	1975
OU1	318.05-04-078.000	12 Roosevelt St	Single Family Residence	1973
OU1	318.05-04-079.000	10 Roosevelt St	Single Family Residence	1975
OU2	318.05-04-080.000	8 Roosevelt St	Single Family Residence	1973
OU2	318.05-04-081.000	4 Roosevelt St	Single Family Residence	1974
OU2	318.05-04-082.000	123 Corning Blvd	Single Family Residence	1976
OU2	318.05-04-083.000	125 Corning Blvd	Single Family Residence	1975
OU2	318.05-04-084.000	127 Corning Blvd	Single Family Residence	1973
OU5	300.17-01-013.000	179 E Pulteney St	Single Family Residence	1930
OU5	300.17-01-014.000	181 E Pulteney St	Single Family Residence	1930
OU5	300.17-01-015.000	3 High St	Multi Family Residence	1896
OU5	300.17-01-017.000	5 High St	Single Family Residence	1860
OU5	300.17-01-018.000	9 High St	Single Family Residence	1947
OU5	300.17-01-019.000	11 High St	Single Family Residence	1972
OU5	300.17-01-020.000	13 High St	Single Family Residence	1950
OU5	300.17-01-022.000	15 High St	Single Family Residence	1989
OU5	300.17-01-023.000	17 High St	Single Family Residence	1946
OU5	300.17-01-024.000	37 High St	Single Family Residence	1973
OU5	300.17-01-025.000	39 High St	Single Family Residence	1975
OU5	300.17-01-026.000	43 High St	Single Family Residence	1948
OU5	300.17-01-030.000	44 High St	Single Family Residence	1973
OU5	300.17-01-031.000	42 High St	Single Family Residence	1953
OU5	300.17-01-033.000	38 High St	Single Family Residence	1942
OU5	300.17-01-034.000	34 High St	Single Family Residence	1940
OU5	300.17-01-035.000	28 High St	Single Family Residence	1961
OU5	300.17-01-036.000	24 High St	Single Family Residence	1951
OU5	300.17-01-037.000	22 High St	Single Family Residence	1940
OU5	300.17-01-038.000	14 High St	Single Family Residence	1945
OU5	300.17-01-039.000	6 High St	Single Family Residence	1945
OU5	300.17-01-040.000	4 High St	Single Family Residence	1942
OU5	300.17-01-042.000	High St	Residential - Vacant Land	--
OU5	300.17-01-043.000	201 E Pulteney St	Single Family Residence	1915
OU5	300.17-01-044.000	203 E Pulteney St	Single Family Residence	1915
OU5	300.17-01-045.000	205 E Pulteney St	Single Family Residence	1925
OU5	300.17-01-046.000	207 E Pulteney St	Single Family Residence	1945
OU5	300.17-01-047.000	Earl St	Residential - Vacant Land	--
OU5	300.17-01-048.000	15 Earl St	Single Family Residence	1950
OU5	300.17-01-049.100	21 Earl St	Single Family Residence	1920
OU5	300.17-01-049.200	17 Earl St	Residential - Vacant Land	--
OU5	300.17-01-050.000	23 Earl St	Single Family Residence	1950
OU5	300.17-01-051.000	25 Earl St	Multi Family Residence	1960
OU5	300.17-01-052.000	27-29 Earl St	Multi Family Residence	1920
OU5	300.17-01-053.000	31 Earl St	Single Family Residence	1955
OU5	300.17-01-054.000	33 Earl St	Single Family Residence	1950
OU5	300.17-02-003.000	34 Earl St	Single Family Residence	1952
OU5	300.17-02-004.000	32 Earl St	Single Family Residence	1976
OU5	300.17-02-005.000	30 Earl St	Single Family Residence	1974
OU5	300.17-02-006.000	26 Earl St	Single Family Residence	1971
OU5	300.17-02-007.000	22 Earl St	Single Family Residence	1940
OU5	300.17-02-008.000	20 Earl St	Single Family Residence	1960
OU5	300.17-02-009.000	18 Earl St	Single Family Residence	1976
OU5	300.17-02-010.000	16 Earl St	Single Family Residence	1940
OU5	300.17-02-011.000	12 Earl St	Single Family Residence	1939
OU5	300.17-02-012.000	211 E Pulteney St	Single Family Residence	1940
OU5	300.17-02-013.000	215 E Pulteney St	Single Family Residence	1940
OU5	300.17-02-014.000	223 E Pulteney St	Single Family Residence	1940
OU5	300.17-02-015.000	9 Clara St	Single Family Residence	1915
OU5	300.17-02-016.000	11 Clara St	Single Family Residence	1955
OU5	300.17-02-017.000	13 Clara St	Single Family Residence	1955
OU5	300.17-02-018.000	15 Clara St	Single Family Residence	1960
OU5	300.17-02-019.000	17 Clara St	Single Family Residence	1952
OU5	300.17-02-020.000	19 Clara St	Single Family Residence	1978
OU5	300.17-02-025.000	18 Clara St	Single Family Residence	1973
OU5	300.17-02-026.000	16 Clara St	Single Family Residence	1951
OU5	300.17-02-027.000	14 Clara St	Single Family Residence	1951



Table 3-1
Property Information for OU1, OU2 and OU5
Study Area, Corning, New York
(continued)

OU	Parcel ID	Address	Property Classification ¹	Year Built
OU5	300.17-02-028.000	12 Clara St	Single Family Residence	1975
OU5	300.17-02-029.000	Clara St	Residential - Vacant Land	--
OU5	300.17-02-030.000	233 E Pulteney St	Single Family Residence	1946
OU5	300.17-02-031.000	235 E Pulteney St	Single Family Residence	1949
OU5	300.17-02-032.000	237 E Pulteney St	Single Family Residence	1952
OU5	300.17-02-033.000	239 E Pulteney St	Single Family Residence	1925
OU5	300.17-02-034.000	1 James St	Single Family Residence	1937
OU5	300.17-02-035.000	3 James St	Single Family Residence	1973
OU5	300.17-02-036.000	5 James St	Multi Family Residence	1979
OU5	300.17-02-037.000	7 James St	Single Family Residence	1974
OU5	300.17-02-038.000	9-11 James St	Multi Family Residence	1978
OU5	300.17-02-040.100	4 James St	Single Family Residence	1999
OU5	300.17-02-040.200	8 James St	Single Family Residence w/Apartment	2000
OU5	300.17-02-041.000	241 E Pulteney St	Single Family Residence	1900
OU5	300.17-02-042.000	243 E Pulteney St	Single Family Residence	1900
OU5	300.17-02-043.000	245 E Pulteney St	Single Family Residence	1900
OU5	300.17-02-044.000	247 E Pulteney St	Single Family Residence	1900
OU5	300.17-02-047.100	249 E Pulteney St	Single Family Residence	1895
OU5	300.17-02-049.000	253 E Pulteney St	Single Family Residence	1953
OU5	300.17-02-050.000	255 E Pulteney St	Single Family Residence	1830
OU5	300.17-02-051.000	257 E Pulteney St	Single Family Residence	1974
OU5	300.17-02-053.000	259 E Pulteney St	Single Family Residence	1935
OU5	300.17-02-054.000	261-263 E Pulteney St	Multi Family Residence	1988
OU5	300.17-02-055.000	267 E Pulteney St	Single Family Residence	1951
OU5	300.17-02-056.000	E Pulteney St (ROW to dyke)	Residential - Vacant Land	--
OU5	300.17-02-057.000	271 E Pulteney St	Residential - Vacant Land	--
OU5	318.05-02-011.000	170 South Pl	Single Family Residence	1949
OU5	318.05-02-012.000	174 South Pl	Single Family Residence	1949
OU5	318.05-02-013.000	178 South Pl	Single Family Residence	1946
OU5	318.05-02-014.000	182 South Pl	Single Family Residence	1946
OU5	318.05-02-015.000	186 South Pl	Single Family Residence	1950
OU5	318.05-02-016.000	33 Pyrex St	Single Family Residence	1950
OU5	318.05-02-017.000	37 Pyrex St	Single Family Residence	1947
OU5	318.05-02-018.000	185 South Pl	Single Family Residence	1948
OU5	318.05-02-019.000	183 South Pl	Single Family Residence	1948
OU5	318.05-02-020.000	179 South Pl	Single Family Residence	1947
OU5	318.05-02-021.000	175 South Pl	Single Family Residence	1940
OU5	318.05-02-027.000	174 North Pl	Single Family Residence	1950
OU5	318.05-02-028.000	178 North Pl	Single Family Residence	1948
OU5	318.05-02-029.000	182 North Pl	Single Family Residence	1950
OU5	318.05-02-030.000	186 North Pl	Single Family Residence	1950
OU5	318.05-02-031.000	41 Pyrex St	Single Family Residence	1945
OU5	318.05-02-032.000	45 Pyrex St	Single Family Residence	1947
OU5	318.05-02-033.000	187 North Pl	Single Family Residence	1945
OU5	318.05-02-034.000	183 North Pl	Single Family Residence	1946
OU5	318.05-02-035.000	179 North Pl	Single Family Residence	1946
OU5	318.05-02-036.000	175 North Pl	Single Family Residence	1946
OU5	318.05-02-041.000	174 E Pulteney St	Single Family Residence	1950
OU5	318.05-02-042.000	178 E Pulteney St	Single Family Residence	1946
OU5	318.05-02-043.000	182 E Pulteney St	Single Family Residence	1948
OU5	318.05-02-044.000	186 E Pulteney St	Single Family Residence	1947
OU5	318.05-02-045.000	47-57 Pyrex St	Apartment	--

¹ All properties within OU1, OU2, and OU5 are zoned R1 (low density residential)

Notes:

--- = Not available

OU1 - Operable Unit 1 (Residential Area)

OU2 - Operable Unit 2 (Residential Area at the Eastern End of Corning Boulevard)

OU5 - Operable Unit 5 (Residential Expansion Area)



4. PRE-DESIGN INVESTIGATION ACTIVITIES

As described in Section 2.3, soil boring and surface soil sampling activities have been performed in OU1, OU2 and OU5 in 2014 and 2015. This section describes the sampling planned under this PDI Work Plan. Consistent with the objectives stated in Subsection 1.1, this sampling will support the design of remediation providing for excavation and removal of Subject Material to a depth of up to 2 ft bgs with a soil cover.

The subsections below describe the field investigation methodologies to be utilized for the PDI Work Plan activities in OU1, OU2 and OU5. The methodologies may be adjusted in the field based upon a variety of factors including field conditions, selected subcontractor equipment and other necessary adjustments. Corning Incorporated will notify NYSDEC of any proposed significant changes or deviations from the approved PDI Work Plan (including any proposed use of investigation methodologies other than those described below), and NYSDEC approval will be obtained prior to implementation of significant changes. Corning Incorporated will verbally discuss minor field adjustments that do not affect the project objectives with the NYSDEC project manager and confirm such discussions by subsequent email and/or documentation in the field notes.

Planned PDI Work Plan activities include a combination of soil boring and surface soil sampling. The number of sampling locations, specific to each OU, is described in the subsequent subsections. Final locations will be established based on utility clearance, accessibility, and discussions with property owners/lessees. SOPs for sample collection, handling and shipment are provided in Appendix D.

4.1 WRITTEN ACCESS CONSENT

Property within OU1 and OU5 is not owned by or under the control of Corning Incorporated or the NYSDEC. Therefore, individual property owners will need to agree to access by Corning Incorporated prior to the field investigation. It is expected that some sampling locations may need to be modified as a result of access issues.



Prior to WESTON performing work at each property, and after Corning Incorporated obtains written consent to access, each individual property owner will be notified by WESTON of pending activities on the owner's property.

4.2 FIELD INVESTIGATION METHODOLOGIES

4.2.1 Subsurface Soil Sampling

Generally, either Geoprobe® or hollow-stem auger drilling technologies will be used to install soil borings to characterize the subsurface soils. Where possible, Geoprobe drilling technology will be utilized to minimize the quantity of investigative derived waste (IDW) generated during field activities. A hollow-stem auger drill rig will be utilized to install borings in locations where a Geoprobe cannot penetrate to the desired depth. All drilling locations will be utility cleared prior to drilling. No soil borings will be advanced through driveways, sidewalks, patios, or other similar property features. Soil boring locations will be recorded by a New York State-licensed surveyor or by using a hand-held global positioning system (GPS) with sub-meter accuracy.

At each Geoprobe boring location, soil sampling will (if possible) be conducted on a continuous basis from the ground surface to the maximum depth using a 2-inch diameter, 4-foot long macrocore sampler. Retrieved soil samples will be examined in the field for physical description by a qualified WESTON geologist and screened using a photoionization detector (PID). Layers of fill material containing ash, brick and/or glass will be identified in the field by the WESTON geologist. A layer of fill material is defined as a non-native material containing ash, brick and/or glass with a thickness greater than 1 inch. All Geoprobe rods and associated drilling equipment will be cleaned between boring locations using the procedures described in Appendix D.

Where hollow-stem auger drilling technology is used, hollow-stem augers will be extended from ground surface to the desired depth. Samples will be continuously collected with a 2-foot long split-spoon sampler during drilling for physical description by a qualified WESTON geologist in the field and screened with a PID. Layers of fill material containing ash, brick and/or glass will be identified in the field by the WESTON geologist. All hollow-stem augers and associated drilling equipment will be cleaned between boring locations using the procedures described in Appendix D.



Samples will be visually examined and a description prepared by a qualified WESTON geologist in accordance with the procedure described in Appendix D. The description will generally be prepared using the Unified Soil Classification System (ASTM D2487, 2011), and will include color, moisture content, texture, layering, etc. Any non-native material present in the sample will be noted and described (type, color, texture, moisture content, etc.) and any layer of fill material containing ash, brick and/or glass will be noted in the field logs. Descriptions of the collected samples will be recorded in the field log book or soil boring log form. Photographs of the soil cores will be taken.

All non-dedicated sampling equipment will be decontaminated by washing with phosphate-free detergent and rinsed with distilled water prior to and between sampling locations. Decontamination procedures are described in Appendix D (Standard Operating Procedures). Alternatively, dedicated, disposable sampling equipment (e.g., scoops, plastic blending trays) may be used.

Soil samples and appropriate quality control (QC) samples (e.g., duplicate samples) will be collected from the sampling cores, homogenized, placed in appropriate sample containers, in iced coolers and shipped with completed chain-of-custody documentation to TestAmerica Laboratories, Inc. in Buffalo, New York (TestAmerica) for analysis. The quantity and types of samples to be collected from each boring are discussed in Section 4.3.

All boreholes will be backfilled with a cement/bentonite grout mixture using a tremie rod, or by placing dry bentonite pellets in the borehole followed by water to hydrate them in place. The surface will be restored with appropriate material (i.e., soil). IDW from this investigation will be contained in sealed containers (e.g., drums or other appropriate containers) and staged in a secondary containment area at the designated, NYSDEC-approved, staging area, located outside the Study Area, pending proper disposal.

4.2.2 Surface and Shallow Soil Sampling

Surface soil samples will be collected for analysis from 0 to 2 in bgs excluding the vegetative cover or sod layer. Shallow soil samples will be collected from 2 in bgs to 2 ft bgs excluding the vegetative cover or sod layer. Prior to sample collection, visible vegetative matter (i.e., sod layer)



will be removed. Surface soil and shallow soil samples will be collected using a small Geoprobe rig, a hand-held steel soil auger, or a hand-held stainless steel scoop. The soil will be described, noting the color, moisture content, texture, layering, evidence of disturbance (foreign debris), and the distribution/abundance of roots. If present, layers of fill material containing ash, brick and/or glass will be noted in the field logs. Surface and shallow soil samples will be homogenized and placed directly into appropriate sample containers.

Surface soil and shallow soil sample locations will be recorded by a New York State-licensed surveyor or by using a hand-held GPS with sub-meter accuracy. All non-dedicated sampling equipment will be decontaminated by washing with phosphate-free detergent and rinsing with distilled water prior to and between sampling locations. Alternately, dedicated, disposable sampling equipment (e.g., scoops, plastic blending trays) may be used. Decontamination fluids will be collected and contained in sealed containers (e.g., drums or other appropriate containers) and staged in a secondary containment area at the designated, NYSDEC-approved, staging area, located outside the Study Area, pending proper disposal.

Soil samples and appropriate QC samples (e.g., duplicate samples) will be placed in appropriate sample containers, in iced coolers and shipped with completed chain-of-custody documentation to TestAmerica for analysis.

4.2.3 Analytics

A summary of planned sampling activities including the number of samples and anticipated analyses is provided in Table 4-1. Detailed descriptions of the sampling approach and rationale are included in Section 4.3 of this PDI Work Plan. The analytical methods/protocols to be used during this project as well as the expanded list of parameters for analysis are provided in Table 4-2 and Table 4-3, respectively.

4.2.4 Quality Assurance / Quality Control

To ensure quality throughout the project, trained and experienced personnel will be utilized, and SOPs and analytical methods for sample collection, preservation, analysis, and documentation will be followed, consistent with previous sampling in the Study Area.



In addition to the laboratory quality assurance (QA) and QC samples analyzed in accordance with the laboratory QA/QC Plan, several types of field QC samples will be collected and submitted for analysis during the course of the field investigation activities to assess the quality of the data resulting from the field sampling program. These samples include:

- **Duplicates:** These samples are duplicate samples collected in the field and submitted to the laboratory without indication of the corresponding parent sample. These samples will be collected at a rate of one per every 20 samples and will provide a measure of laboratory precision and matrix variability.
- **Field Rinsate Blanks:** These samples will be collected to document the adequacy of field decontamination of reusable sampling equipment. Field rinsate blanks will be prepared by pouring deionized water over the sampling equipment after a decontamination procedure has been completed. This rinse water is then collected and submitted for analysis to provide an indication of the effectiveness of decontamination procedures (carry-over from sample to sample). These samples will be prepared at a rate of one per 20 samples.

The number of QA/QC samples anticipated is tabulated in Table 4-1. Further description of the QA/QC samples and analytical procedures are provided in the QAPP provided in Appendix C.

Laboratory data deliverable packages will meet the requirements of NYSDEC Analytical Services Protocol (ASP) Category B (See DER-10 Appendix 2B Section 1.0b). Validation of laboratory data deliverable packages will be performed as described in Section 5.2.4.

4.2.5 Survey Activities

Property surveys will be conducted at the residential properties, by a New York State-licensed surveyor, prior to, or during, sampling activities. The surveyor will locate property lines in the field and record locations of physical features (e.g. driveways, walkways, decks, etc.). Surface soil, shallow soil, and soil boring locations will be recorded by the New York State-licensed surveyor or by using a hand-held GPS unit with sub-meter accuracy.

4.2.6 Waste Handling

All non-dedicated sampling and monitoring equipment will be decontaminated by washing with phosphate-free detergent and rinsing with distilled water, or through other approved decontamination methods. All rinse water will be containerized and properly disposed by Corning



Incorporated in accordance with applicable requirements. Decontamination procedures are described in Appendix D (Standard Operating Procedures).

All soil and water IDW will be handled in accordance with DER-10 Section 3.3(e). Drill cuttings and other soil and water generated during investigation activities will be collected and containerized in sealed containers (e.g., drums or other appropriate containers) daily. The filled containers will be staged in a secondary containment area at a designated location outside the Study Area approved by NYSDEC, pending proper disposal. The IDW will be properly disposed by Corning Incorporated in accordance with applicable requirements.

4.3 INVESTIGATION ACTIVITIES

The planned sampling contained in this PDI Work Plan supports the proposed remediation activities on a property-by-property basis. Samples will be collected for characterization purposes and final endpoint confirmation purposes, so they will be analyzed by a New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP)-certified laboratory, in accordance with the QAPP provided in Appendix C.

Due to the confidential nature of sample collection on private properties, no sampling location maps are included in the PDI Work Plan. Sample locations are described in tabular form in Table 4-4. Individual properties are identified by property codes, and sampling locations are described using property features and/or previous sampling locations.

For ease of implementation, the type of sampling planned in this PDI Work plan is divided into five categories. The sampling category dictates both the type of sample to be collected and additional steps to be taken based on field observations (e.g. if step-out borings are necessary). These five sampling categories are described in more detail in the following subsections.

4.3.1 Category A Characterization Samples

Category A samples are being collected to achieve a total of at least four surface soil samples and two soil borings on each residential property that is not included in the expedited sampling



program¹. Category A soil borings will be installed prior to the collection of Category A surface soil samples on each property.

Category A soil borings will be advanced at properties identified in Table 4-4 via Geoprobe to 15 ft bgs using methods described in Section 4.2.1 and in Appendix D (Standard Operating Procedures) of this PDI Work Plan. Samples will be collected from the soil boring using a 2-inch diameter, 4-foot-long, macrocore sampler in accordance with Section 4.2.1 of this PDI Work Plan. Generally one of these soil borings will be advanced in the front yard and one in the backyard, where possible, at locations for which property owners report that they have observed the presence, if any, of ash, brick and/or glass.

In soil borings where no layer of fill material is encountered, up to three soil samples will be collected; one from the soil at 0 to 2 in bgs excluding the ground cover or sod layer, one from the soil at 2 in to 2 ft bgs and one sample of the native material in the shallowest interval it occurs. If native soil is encountered in the 0 to 2 ft bgs, two soil samples will be collected per boring; one from the soil at 0 to 2 in bgs excluding the ground cover or sod layer and one from the soil at 2 in to 2 ft bgs. Samples will be analyzed for Target Analyte List (TAL) metals plus mercury.

If a layer of fill material is encountered during drilling of the borings, up to five soil samples will be collected per boring; one from the soil at 0 to 2 in bgs excluding the ground cover or sod layer, one from 2 in to 2 ft bgs (if possible); one from the layer of fill material at depths less than 2 ft bgs (if present); one from the layer of fill material at depths greater than 2 ft bgs (if present); and one from the native material beneath the layer of fill material. Samples will be analyzed for TAL metals plus mercury and Target Compound List (TCL) semi-volatile organic compounds (SVOCs).

Additionally, if a layer of fill material containing ash, brick and/or glass is encountered in a Category A soil boring, additional delineation soil borings will be advanced. These additional soil borings will be advanced as needed to horizontally delineate the layer of fill material containing

¹ 66 properties in OU1 which have been included in the expedited sampling plan required by NYSDEC.



ash, brick and/or glass on the property, and will be advanced and sampled using the procedures for Category B endpoint soil borings described in Section 4.3.2.

Category A surface soil samples will be collected from the 0 to 2 in bgs interval, excluding the sod layer, as indicated in Table 4-4. The locations of the surface soil samples will be determined in the field and may be biased toward areas with the highest potential for soil contact by residents, such as entrance areas, swing sets and gardens. The grass and root layer, if present, will be removed from the sampling interval prior to sample collection, and will be replaced once sample collection has been completed at each location, if possible.

Category A surface soil samples will be analyzed for COPCs. If a layer of fill material containing ash, brick and/or glass was encountered in any of the soil borings advanced on the property, the Category A surface soil samples at that property will be analyzed for TAL metals plus mercury and SVOCs. Additionally, if ash, brick and or glass is observed in a Category A surface soil sample, the sample will be analyzed for TAL metals plus mercury and SVOCs.

4.3.2 Category B Endpoint Samples

Category B endpoint samples are surface soil and/or soil boring samples to be collected at discrete locations to delineate the presence of a specific constituent(s) detected at concentrations greater than SCOs and/or to delineate the horizontal extent of a layer of fill material containing ash, brick and/or glass. Category B endpoint sampling locations, identified in Table 4-4, will be placed approximately 10 to 30 feet from sampling locations where constituents were previously detected at concentrations greater than SCOs and/or from where a layer of fill material containing ash, brick and/or glass was observed. Category B endpoint surface and/or soil boring samples will be collected in accordance with Table 4-4 using methods described in Section 4.2.1, Section 4.2.2 and Appendix D (Standard Operating Procedures) of this PDI Work Plan.

Category B endpoint surface soil samples will be collected from the 0 to 2 in bgs interval, excluding the sod layer, and analyzed for TAL metals plus mercury and SVOCs.

Category B soil borings will be advanced via Geoprobe to depths of up to 15 ft bgs using methods described in Section 4.2.1 and in Appendix D (Standard Operating Procedures) of this PDI Work Plan. If no layer of fill material containing ash, brick and/or glass is encountered in the Category



B soil borings, up to three soil samples will be collected as endpoint confirmation samples as indicated in Table 4-4: one sample from the soil at 0 to 2 in bgs excluding the ground cover or sod layer, one sample from 2 in to 2 ft bgs, and one sample of the native material in the shallowest interval it occurs. Samples will be analyzed for TAL metals plus mercury and SVOCs.

If a layer of fill material is encountered in Category B soil borings no soil samples will be collected and additional 'step-out' soil borings will be advanced approximately 10 to 15 feet from the previous sampling location. However, if previous sampling and the presence of multiple soil borings in an area indicate the presence of a layer of fill material containing ash, brick and/or glass, a larger 30 foot spacing may be used. This process of advancing additional step-out soil borings, but not collecting soil samples, will continue until: (1) a soil boring is advanced and no layer of fill material containing ash, brick and/or glass is encountered, (2) a property boundary and/or property structure is reached, or (3) the sampling encroaches onto previous soil boring locations where information about that area is known.

Endpoint confirmation samples will be collected from soil borings advanced at a minimum of every 30 linear feet around a delineated area of a layer of fill material containing ash, brick and/or glass except where a property boundary or property structure is reached. Category B endpoint confirmation samples will consist of two soil samples per boring: one from the soil at 0 to 2 in bgs excluding the ground cover or sod layer, and one from 2 in to 2 ft bgs. Endpoint confirmation samples will be analyzed for TAL metals plus mercury and SVOCs.

In cases where the layer of fill material containing ash, brick and/or glass is less than 2 feet deep, soil samples may be collected beneath the layer of fill material, but shallower than 2 feet bgs, as confirmation samples for an excavation less than 2 ft bgs. Samples will be collected at 1 per 900 square feet (30 ft by 30 ft grid) and will be analyzed for TAL metals plus mercury and SVOCs.

If field observations appear to indicate a layer of fill material containing ash, brick and/or glass extends from an individual property to an adjacent property within the Study Area, the WESTON Field Team Leader or Project Manager will verbally notify NYSDEC's representative present in the Study Area or NYSDEC's Project Manager. If NYSDEC determines that additional investigations are required on the adjacent property within the Study Area, NYSDEC will notify the property owner of such adjacent property and determine if the property owner is willing to



grant written consent of access for the work. Within a week of NYSDEC's notifying Corning in writing that the property owner is willing to grant written consent to access, Corning will mail an agreement seeking consent to access to the property owner, to perform sampling as discussed in this PDI Work Plan. The field work will be scheduled promptly upon Corning's receipt of the written consent for access, signed by the property owner.

4.3.3 Category C Soil Cover Confirmation Soil Borings

Category C soil cover confirmation soil borings will be advanced on an approximately 30 foot grid to verify the protectiveness of the existing soil cover in areas where previous sampling indicated the presence of Subject Material at depths greater than 2 ft bgs.

Category C soil cover confirmation soil borings will be advanced via Geoprobe to the depth of 2 ft bgs using methods described in Section 4.2.1 and in Appendix D (Standard Operating Procedures) of this PDI Work Plan. If no layer of fill material containing ash, brick and/or glass is encountered, soil samples will be collected and analyzed for TAL metals plus mercury and SVOCs.

If a layer of fill material containing ash, brick and/or glass is encountered in the 0 to 2 ft bgs interval in Category C soil cover confirmation soil borings, no soil samples will be collected and additional delineation soil borings will be advanced. The additional soil borings will be advanced as needed to horizontally delineate the layer of fill material containing ash, brick and/or glass on the property and will be advanced and sampled using the procedures for Category B endpoint confirmation soil borings described in Section 4.3.2.

4.3.4 Category D Characterization Samples

Category D characterization samples, identified in Table 4-4, are surface soil and/or soil boring samples, requested by NYSDEC, to collect additional analytical data. Category D surface and/or soil boring samples will be collected in accordance with Table 4-4 and analyzed for TAL metals plus mercury and SVOCs using methods described in Section 4.2.1, Section 4.2.2 and Appendix D (Standard Operating Procedures) of this PDI Work Plan. If no layer of fill material containing ash, brick and/or glass is encountered, soil samples will be collected in accordance with Table 4-4 and analyzed for TAL metals plus mercury and SVOCs.



Category D characterization surface soil samples will be collected from the 0 to 2 in bgs interval, excluding the sod layer, as indicated on Table 4-4. The samples will be analyzed for TAL metals plus mercury and SVOCs.

If a layer of fill material containing ash, brick and/or glass is encountered in a Category D characterization soil boring, no soil samples will be collected and additional delineation soil borings will be advanced. These additional soil boring will be advanced as needed to horizontally delineate the layer of fill material containing ash, brick and/or glass on the property and will be advanced and sampled using the procedures for Category B endpoint soil borings described in Section 4.3.2.

4.3.5 Category E Samples

Category E samples, identified in Table 4-4, are surface soil and/or soil boring samples that will be collected at previous sampling locations to supplement existing analytical data so that the sample location can be used an endpoint sample or as a soil cover sample. Endpoint samples and soil cover samples will be analyzed for TAL metals plus mercury and SVOCs. Category E samples will be collected at previous sampling locations and depth intervals identified in Table 4-4, where the previous analysis did not include all of these parameters.

Surface soil and/or soil boring samples will be collected and analyzed in accordance with Table 4-4 to supplement existing analytical data to achieve complete analysis of all TAL metals plus mercury and SVOCs at these sample locations. It should be noted the samples will be analyzed for only the parameters that were not previously analyzed at the sampling location.

If a layer of fill material containing ash, brick and/or glass is encountered in a Category E soil boring, no soil samples will be collected and additional delineation soil borings will be advanced. These additional soil borings will be advanced as needed to horizontally delineate the layer of fill material containing ash, brick and/or glass on the property and will be advanced and sampled using the procedures for Category B endpoint soil borings described in Section 4.3.2.

4.3.6 Additional Investigation Samples

Based on preliminary analytical results, additional delineation sampling may be conducted, as needed, to horizontally delineate the limits of an area with concentrations greater than SCOs.



During this additional round of sampling, surface soil or soil boring samples will be collected using the procedures described above and will be analyzed for the analytical compounds detected at concentrations greater than SCOs in the preliminary results.

4.4 IN SITU DISPOSAL PROFILE SAMPLING

Once Corning Incorporated has delineated a remediation area and it has been approved by NYSDEC, sampling for disposal profiling may be conducted prior to remedial activities. This sampling will include collection of *in situ* samples for a representative disposal profile sample(s) in areas designated for remediation, prior to excavation activities, to facilitate landfill pre-acceptance of material that will be excavated and direct loaded for disposal.

Disposal profile sampling will involve the collection of *in situ* composite samples and *in situ* discrete samples prior to excavation activities. *In situ* composite disposal profile samples will be collected at a rate of approximately one composite sample for the first 100 cubic yards, and then one sample per every 300 cubic yards of Subject Material to be excavated, with a minimum of one sample per property. Each *in situ* composite disposal profile sample will consist of subsamples, collected from the depth interval to be excavated, excluding the ground cover if any, at 3 to 5 locations from within the planned remediation area, mixed together as one composite sample. *In situ* discrete disposal profile samples will be collected at a rate of approximately one sample for the first 100 cubic yards and then one sample per every 300 cubic yards of Subject Material to be excavated, with a minimum of one sample per property. Each *in situ* discrete sample will be collected from the depth interval to be excavated excluding the ground cover if any. All disposal profile samples will be analyzed for Toxicity Characteristic Leaching Procedure (TCLP) Resource Conservation and Recovery Act (RCRA) metals.



SECTION 4

TABLES



**Table 4-1
Sample Count Summary Table**

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis ⁽¹⁾	No. Primary Samples	Estimated No. QC Samples			Total
						DUP	FB	MS/MSD	
SOIL									
OU1 Residential Area	159	Soil Borings	2-5	COPCs	0	0	0	0	0
				TAL Metals + Hg	472	24	24	24	544
				SVOCs	246	13	13	13	285
	207	Surface Soil ⁽²⁾	1	COPCs	40	2	2	2	46
				TAL Metals + Hg	148	8	8	8	172
				TAL Metals + Hg (minus COPCs)	4	1	1	1	7
				Hg only	1	1	1	1	1
				SVOCs	166	9	9	9	193
	251	Shallow Soil ⁽³⁾	1-3	COPCs	0	0	0	0	0
				TAL Metals + Hg	445	23	23	23	514
				SVOCs	502	26	26	26	580
	OU2 Residential Area at the Eastern End of Corning Blvd	8	Soil Borings	2-5	COPCs	0	0	0	0
TAL Metals + Hg					24	2	2	2	30
SVOCs					24	2	2	2	30
29		Shallow Soil ⁽³⁾	1-3	COPCs	0	0	0	0	0
				TAL Metals + Hg	56	3	3	3	65
				TAL Metals + Hg (minus COPCs)	2	1	1	1	5
				SVOCs	58	3	3	3	67
OU5 Residential Area	61	Soil Borings	2-5	COPCs	0	0	0	0	0
				TAL Metals + Hg	175	9	9	9	202
				SVOCs	145	8	8	8	169
	97	Surface Soil ⁽²⁾	1	COPCs	22	2	2	2	28
				TAL Metals + Hg	75	4	4	4	87
				SVOCs	75	4	4	4	87
	210	Shallow Soil ⁽³⁾	1-2	COPCs	0	0	0	0	0
				TAL Metals + Hg	416	21	21	21	479
SVOCs				420	21	21	21	483	
TOTAL SOIL ANALYSES:									4,074

Notes:

⁽¹⁾ Analytical methods are presented in Table 4-2 and complete analyte lists are presented in Table 4-3.

⁽²⁾ Surface samples collected from 0 - 2 inches bgs

⁽³⁾ Shallow soil sample locations include a surface soil sample collected from 0 inches bgs to 2 inches bgs and a shallow soil sample collected from 2 inches bgs to 2 feet bgs

COPCs - Constituents of Potential Concern (i.e., arsenic, cadmium and lead)

TAL - Target Analyte List

SVOC - semi-volatile organic compound

QA/QC - quality assurance/quality control

DUP - duplicate sample

FB - field blank

MS/MSD - matrix spike/matrix spike duplicate

TB - trip blank

No. - number

bgs - below ground surface

NA - not applicable (no analytical samples collected)

NS - not sampled

Assumptions:

All samples will be analyzed for list (as shown)

DUP, FB and MS/MSD will be collected at 20%



Table 4-2
Analytical Methodologies

Analysis	Analytical Methods	Container	Preservation	Hold Time
SOIL				
COPCs	SW846 6010	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	6 months
TAL Metals	SW846 6010	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	6 months
Mercury	SW846 7471	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	28 days
SVOCs	SW846 8270	30 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4°C	14 days

Notes:

COPCs - Constituents of Potential Concern

TAL - Target Analyte List

SVOCs - Semi-volatile Organic Compounds



Table 4-3
Reporting Limits and Method Detection Limits

	Soil	
	RL	MDL
COPCs [Method SW846 6010]	mg/Kg	
Arsenic	2.00	0.400
Cadmium	0.200	0.0300
Lead	1.00	0.240
Metals [Method SW846 6010]	mg/Kg	
Aluminum	10.0	4.40
Antimony	15.0	0.400
Arsenic	2.00	0.400
Barium	0.500	0.110
Beryllium	0.200	0.0280
Boron	2.00	0.190
Cadmium	0.200	0.0300
Calcium	50.0	3.30
Chromium	0.500	0.200
Cobalt	0.500	0.0500
Copper	1.00	0.210
Iron	10.0	1.10
Lead	1.00	0.240
Magnesium	20.0	0.927
Manganese	0.200	0.0320
Nickel	5.00	0.230
Potassium	30.0	20.0
Selenium	4.00	0.400
Silver	0.600	0.200
Sodium	140	13.0
Thallium	6.00	0.300
Vanadium	0.500	0.110
Zinc	2.00	0.153
Mercury [Method SW846 7471B/7470A]	mg/Kg	
Mercury	0.0200	0.00810
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270]	µg/Kg	
Biphenyl	170	28.2
bis (2-chloroisopropyl) ether	170	34.0
2,4,5-Trichlorophenol	170	46.0
2,4,6-Trichlorophenol	170	34.0
2,4-Dichlorophenol	170	18.0
2,4-Dimethylphenol	170	72.7
2,4-Dinitrophenol	1660	784
2,4-Dinitrotoluene	170	35.0
2,6-Dinitrotoluene	170	20.0
2-Chloronaphthalene	170	28.0
2-Chlorophenol	170	31.0
2-Methylphenol	170	20.0
2-Methylnaphthalene	170	34.0
2-Nitroaniline	330	25.0
2-Nitrophenol	170	48.0
3,3'-Dichlorobenzidine	330	200
3-Nitroaniline	330	47.0
4,6-Dinitro-2-methylphenol	330	170
4-Bromophenyl phenyl ether	170	24.0
4-Chloro-3-methylphenol	170	42.0
4-Chloroaniline	170	42.0
4-Chlorophenyl phenyl ether	170	21.0
4-Methylphenol	330	20.0
4-Nitroaniline	330	89.0
4-Nitrophenol	330	159
Acenaphthene	170	25.0
Acenaphthylene	170	22.0
Acetophenone	170	23.0
Anthracene	170	42.0
Atrazine	170	59.0
Benzaldehyde	170	135
Benzo[a]anthracene	170	27.6
Benzo[a]pyrene	170	25.0
Benzo[b]fluoranthene	170	27.0



Table 4-3 (continued)
Reporting Limits and Method Detection Limits

	Soil	
	RL	MDL
<i>Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270] (continued)</i>	µg/Kg	
Benzo[g,h,i]perylene	170	19.0
Benzo[k]fluoranthene	170	22.0
Bis(2-chloroethoxy)methane	170	36.0
Bis(2-chloroethyl)ether	170	22.0
Bis(2-ethylhexyl) phthalate	170	58.0
Butyl benzyl phthalate	170	28.0
Caprolactam	170	51.0
Carbazole	170	20.0
Chrysene	170	38.0
Dibenz(a,h)anthracene	170	30.0
Di-n-butyl phthalate	170	29.0
Di-n-octyl phthalate	170	20.0
Dibenzofuran	170	20.0
Diethyl phthalate	170	22.0
Dimethyl phthalate	170	20.0
Fluoranthene	170	18.0
Fluorene	170	20.0
Hexachlorobenzene	170	23.0
Hexachlorobutadiene	170	25.0
Hexachlorocyclopentadiene	170	23.0
Hexachloroethane	170	22.0
Indeno[1,2,3-cd]pyrene	170	22.0
Isophorone	170	36.0
N-Nitrosodi-n-propylamine	170	29.0
N-Nitrosodiphenylamine	170	138
Naphthalene	170	22.0
Nitrobenzene	170	30.0
Pentachlorophenol	330	170
Phenanthrene	170	25.0
Phenol	170	26.0
Pyrene	170	20.0
<i>TCLP Metals [Method SW846 6010 / 7470]</i>	mg/L	
Arsenic	0.0150	0.00555
Barium	1.00	0.100
Cadmium	0.00200	0.000500
Chromium	0.0200	0.0100
Lead	0.0200	0.00300
Mercury	0.000200	0.000120
Selenium	0.0250	0.00870
Silver	0.00600	0.00170

Notes:

mg/Kg - milligram per kilogram

µg/Kg - microgram per kilogram

mg/L - milligram per liter

COPCs - constituents of potential concern

RL - Reporting Limit

MDL - Method Detection Limit

Listed limits are the highest current MDL and RL inclusive of TestAmerica Buffalo and TestAmerica Edison laboratories for standard analytical testing.



Table 4-4
Pre-Design Investigation Sample Summary

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES001	Category B	3 SB	Around SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around ROWSB041	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southwest of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category E	1 SB	At SB003	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		-	-	Sample location SB006	-	-	-	No action per NYSDEC for SVOCs
OU1	RES002	Category B	1 SB	West of SB006	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	North of SB010	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		-	-	Sample location SS010	-	-	-	No Action per NYSDEC for SVOCs non-detect.
OU1	RES003	Category B	3 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Between SB004 and SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB006	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
OU1	RES004	Category B	2 SB	South and East of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SB	South and West of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU1	RES005	Category E	1 SB	At SB001	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB002	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB003	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	3 SB	Around SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Around SS011	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES007	-	-	Sample location SS002	-	-	-	No action per NYSDEC for arsenic at 16.2 mg/Kg (0-2")
		Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
OU1	RES008	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
		-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES009	-	-	-	-	-	-	No additional sampling
OU1	RES010	Category B	2 SB	North and West of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	ROW	Category B	2 SB	Near RES010SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES012	Category B	1 SB	Southeast of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Southwest of house along property line	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category C	1 SB	Between SB001 and SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES013	Category B	1 SB	Southwest of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southwest of SB005	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category E	1 SB	At SB003	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category E	1 SB	At SB006	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	-
						2"-24"	SVOCs	-
		Category E	1 SB	At SB008	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	-
						2"-24"	SVOCs	-
		Category B	1 SB	Northeast of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB010	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES014	Category B	2 SS	South of SS003, South of SS007	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around SS008/SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB003	2 in bgs	0-2"	SVOCs	-
OU1	RES015	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES016	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES017	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES018	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES019	Category B	4 SB	Around SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS009	2 in bgs	0-2"	SVOCs	-
		Category B	3 SS	Around SS004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES020	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES021	Category B	2 SB	Northeast and Southwest of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB001	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category B	4 SB	Around SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	Northwest of SS001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS006	2 in bgs	0-2"	SVOCs	-
OU1	RES022	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES023	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES024	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES025	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
		Category B	1 SS	Along southwest property line	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU1	RES026	Category B	1 SB	Southwest of SB003	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southeast of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	ROW	Category B	1 SB	Northeast of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES027	Category B	3 SB	Around SB004/SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Southeast and southwest of SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Southwest and southeast of SB005	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Northeast of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Southeast of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES028	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Two additional soil borings required by NYSDEC
OU1	RES029	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES030	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES031	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES032	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES033	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES034	Category B	1 SS	Northeast of SS007/Northwest of SB003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Northeast of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Northeast of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of SS007	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of SS007	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES035	Category D	1 SS	Near addition	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category D	1 SB	Near addition	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
OU1	RES036	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES037	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES038	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES039	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES040	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES042	Category B	1 SB	Southeast of SB004	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	3 SB	Around SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southwest of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		-	-	RES042SB002	-	-	-	Ignore per NYSDEC for Non-Detect SVOCs
OU1	RES043	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES044	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES045	-	-	-	-	-	-	No additional sampling
OU1	RES046	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES047	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES048	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES049	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES050	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES051	-	-	-	-	-	-	NYSDEC No Further Action letter



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES052	Category D	1 SB	Along southern backyard property boundary	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category D	1 SB	Backyard along eastern property boundary	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category B	3 SS	Around SS004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB004	2 in bgs	0-2"	SVOCs	-
		Category B	1 SS	North of SS010	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Southeast and northeast of SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS001	2 in bgs	0-2"	TAL Metals (excluding COPCs), Mercury and SVOCs	-
		Category B	1 SS	South of SB003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of SB003	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category E	1 SB	At SB005	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SS009	2 in bgs	2"-24"	SVOCs	
		Category B	1 SB	Northeast of TP01/SB007	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of SB002	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		-	-	Sample location TP01/SB007	-	-	-	No action per NYSDEC for SVOCs (ND at 4 ft bgs)
OU1	RES055	Category B	1 SB	East of ROWSB042	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Southwest and Southeast of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Northwest and Southwest of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB009	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northwest and southeast of SS002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around SS005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	South of SS009	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northwest and southwest of SS016	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southwest of ROWSB034	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover Soil Sample
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover Soil Sample
		Category E	1 SB	At SB001	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover Soil Sample
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover Soil Sample
		-	-	Sample location SB005	-	-	-	No action per NYSDEC for SVOCs (ND at 2"-24")
		-	-	Sample location SB007	-	-	-	No action per NYSDEC for SVOCs (ND at 2"-24")
OU1	ROW	Category B	1 SB	Southeast of ROWSB034	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Northeast of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of ROWSB042	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES056	Category D	1 SB	Backyard west of pool	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category B	4 SS	Around SS005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB002	2 in bgs	0-2"	SVOCs	-
		Category B	1 SS	Southwest of SS010	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	West and South of SB004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	ROW	Category B	1 SS	Northeast of SS010	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	East of SB004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES057	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2" , 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES058	Category B	1 SB	West of SB006	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SB	West and South of SB001	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	East of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES059	Category B	4 SS	Around SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2" , 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
		-	-	Sample location SS004	-	-	-	No action per NYSDEC for SVOCs
OU1	RES060	-	-	-	-	-	-	No additional sampling
OU1	RES061	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES062	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES063	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES064	-	-	-	-	-	-	No additional sampling



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES067	Category B	1 SB	Along southern property line	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property Soil boring to limit area of institutional control
		Category B	2 SB	North and East of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	ROW	Category D	1 SB	East of RES067	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
OU1	RES068	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES069	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES070	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES071	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES072	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES073	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES074	Category B	4 SB	Around SB001/SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	West of SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS005	2 in bgs	0-2"	SVOCs	-
		Category B	2 SS	North and south of SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB002	2 in bgs	0-2"	SVOCs	-
		Category B	3 SS	Around SS007	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	ROW	Category B	1 SS	West of SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES075	Category B	3 SB	Around SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	East of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB009	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		Category E	1 SB	At SB010	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		Category E	1 SB	At SB008	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		-	-	Sample RES075SB007	-	-	-	Ignore SVOCs non-detect
		-	-	Sample RES075SB006	-	-	-	Ignore SVOCs non-detect
OU1	RES076	-	-	-	-	-	-	No additional sampling
OU1	RES077	Category B	2 SS	Northwest and southwest of SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS001	2 in bgs	0-2"	Mercury	-
		Category B	2 SB	Southwest and Southeast around SB004/SB006	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	RES078	Category B	1 SB	South of SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	East of driveway	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU1	ROW	Category B	1 SB	West of SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES079	Category B	4 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northwest and Southwest of SS007	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Along southeastern property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	1 SB	Southwest of SB006	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category E	1 SB	At SB003	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES080	Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around SS004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB004	2 in bgs	0-2"	SVOCs	-
		Category B	4 SS	Around SS008	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location RES080SB005	-	-	-	No action per NYSDEC for Arsenic at 20.1 mg/kg (2"-24")
OU1	RES081	Category B	4 SB	Around SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	East of SB004 and SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES082	Category B	1 SB	Northeast of ROWSB058/Southwest corner of property	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category E	1 SB	At SB003	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category D	2 SB	Eastern edge of property	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
OU1	ROW	Category B	1 SB	North of ROWSB061	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES083	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	ROW	Category B	1 SB	North of ROWSB043	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES084	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES085	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES086	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES087	Category B	2 SB	Northeast and southwest of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB005	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Cover System Sample
OU1	RES088	Category B	2 SB	Southwest and southeast of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB003	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	South of SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES089	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES090	Category B	3 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	West and North of SB006	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	ROW	Category B	1 SB	South of ROWSB043	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES091	Category B	2 SS	North and south of SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metal, Mercury and SVOCs	Cover System Sample
		Category B	1 SB	East of SB003	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		-	-	Sample location SB006	-	-	-	No action per NYSDEC for cadmium at 3.0 mg/kg (2'-4')
		-	-	Sample location GS001	-	-	-	No action per NYSDEC for arsenic at 17.8 mg/kg (0-12")
OU1	RES092	Category B	3 SS	Around SB002 and SS002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northeast and southwest of SS007	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS003	2 in bgs	0-2"	TAL Metals (excluding COPCs), Mercury and SVOCs	-
		Category B	2 SS	Northeast and southeast of SS001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Northeast of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category C	1 SB	South of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
Category E	1 SB	At SB005	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Cover System Sample		
OU1	RES093	-	-	-	-	-	NYSDEC No Further Action letter	
OU1	RES094	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	ROW	Category B	1 SB	South of ROWSB029	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES095	Category B	2 SB	North and South of SB001/SB005	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	North and East of SS002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	East and west of SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	ROW	Category B	1 SB	North of ROWSB028	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES096	Category B	1 SB	North of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	North and East of SS005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location SB007	-	-	-	No action per NYSDEC for SVOCs at ND (2"-24")
OU1	RES097	Category B	1 SB	Northeast of SB004	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB003	15 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample Soil boring to limit area of institutional control



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES098	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES099	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES100	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES101	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES102	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES103	Category B	2 SB	Southeast and southwest of SB007	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample locations SS001 and SS006	-	-	-	No action per NYSDEC for arsenic at 16.6 and 16.9 mg/kg (0-2")
OU1	ROW	Category B	1 SB	Southwest of SB008	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES104	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES105	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES106	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES107	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES108	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES109	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
		Category B	1 SB	East of barn	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU1	RES110	Category D	1 SB	Near garden	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category C	2 SB	Northwest and southeast of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	2 SB	Northeast and Northwest of SB001 (near house)	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	RES111	Category D	1 SB	Backyard, northwest of house	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category B	2 SB	Along northwest property line	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU1	RES112	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES113	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES114	Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	2 SB	Center of backyard & Northeast corner of house	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	1 SB	North of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB003	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Cover System Sample
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES115	-	-	-	-	-	-	No Additional Sampling
OU1	RES116	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES117	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES118	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES119	-	-	-	-	-	-	No Additional Sampling
OU1	RES120	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES121	Category B	1 SB	Backyard, behind garage	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category D	1 SB	Northwest of ROWTP05SB01	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Required by NYSDEC
OU1	RES122	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES123	Category B	2 SB	Northwest and South of SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Southwest and South of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category D	2 SB	Backyard near addition	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category E	1 SB	At SB005	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category E	1 SB	At SB009	2 ft bgs	0-2" 2"-24"	TAL Metals, Mercury and SVOCs SVOCs	Soil boring for endpoint and to limit area of institutional control
OU1	RES124	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES125	Category B	2 SB	Northeast and Southwest of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Northeast and Southwest of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU1	RES126	-	-	-	-	-	-	NYSDEC No Further Action letter



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES127	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES128	Category B	1 SB	Along northwest property line	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	4 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES129	Category B	1 SB	Northeast of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB003	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	-
						2"-24"	SVOCs	
OU1	RES130	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES131	Category B	1 SB	Northwest of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Northwest and Southeast of SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Between SB002 and SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	1 SS	Southwest of SS003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category C	3 SB	West of house	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB002	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
OU1	ROW	Category B	1 SB	Southwest of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of ROWSB039	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES132	Category B	3 SB	Around SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Northeast corner of property	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category C	1 SB	Backyard near northeast side of house	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category C	2 SB	Front yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SS003	2 in bgs	0-2"	TAL Metals (excluding COPCs), Mercury and SVOCs	-
		Category B	1 SS	Southeast of SS009	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around SS011	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB002	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB001	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB003	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
		Category E	1 SB	At SB004	2 ft bgs	0-2" and 2"-24"	SVOCs	Cover System Sample
OU1	RES133	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES134	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES135	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES136	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES137	Category B	1 SB	Center of backyard	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SS	Along eastern property line	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category C	3 SB	Along western property line	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB002	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		Category E	1 SB	At SB003	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES138	Category E	1 SS	At SB004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	Cover System Sample
		Category B	3 SS	Around SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northeast and Northwest of SB002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB008	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At SB009	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
						2"-24"	SVOCs	Cover System Sample
		Category C	1 SB	Backyard west of garage	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES139	Category A	2 SB	To be determined in the field	15 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	-
						0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	Cover System Sample



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES140	Category B	1 SB	Northeast corner of property	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	2 SB	Southeast and southwest of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SB	North and South of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Between SB006 and SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES141	Category B	1 SB	Northeast of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	South and Southeast of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southeast of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Northeast corner of property	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		-	-	Sample SB001	-	-	-	No action per NYSDEC for SVOCs at ND (2"-24")
OU1	RES142	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES143	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES144	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES145	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES146	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES147	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES148	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES149	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES150	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES151	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES152	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES153	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES154	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES155	-	-	-	-	-	-	No Additional Sampling
OU1	RES156	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES157	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES158	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES159	Category B	1 SB	Around East of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	South and West of SB004	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU1	ROW	Category B	1 SB	North RES159SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES160	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES161	Category B	3 SB	Around SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Northeast corner of property	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category C	2 SB	Center of backyard and East side yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES162	Category B	1 SB	Southeast of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southwest of SS006/SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	South of SB003	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of SB006	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	North of SS005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around SB007	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES163	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES164	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES165	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES166	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES167	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES168	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES169	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES170	Category E	1 SS	At SB003	2 in bgs	0-2"	SVOCs	-
		Category B	2 SS	Northwest and northeast of SS002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES171	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU1	RES172	-	-	-	-	-	-	Property included in Expedited Sampling Plan



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES173	Category B	3 SB	Around SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Northeast and Southwest of SB003	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	3 SB	Around of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	ROW	Category B	2 SB	East and West of ROWSB026	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES174	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES175	Category D	1 SB	East portion of yard, south of SB005	15 ft bgs	2'-4'	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		Category E	1 SS	At SB002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SB004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	RES176	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES177	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES178	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES179	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES180	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES181	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES182	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES183	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES184	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES185	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES186	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES187	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES188	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES189	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES190	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES191	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES192	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES193	Category B	3 SB	Around SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category D	1 SB	Near back deck	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
OU2	RES194	Category B	2 SB	North and East of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SB	East and South of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	North and West of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU1	RES196	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES197	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES198	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES199	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES200	Category B	4 SS	Around SB003	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SB001	2 in bgs	0-2"	SVOCs	-
		Category E	1 SS	At SS007	2 in bgs	0-2"	SVOCs	-
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	Southwest of SS001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	East and South of SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU1	RES201	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES202	-	-	-	-	-	-	NYSDEC No Further Action letter
OU2	RES203	Category B	3 SB	Around SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	12 SB	Across Side and Back Yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU2	ROW	Category B	1 SB	South of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES204	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES205	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES206	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES207	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES208	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES209	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES210	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES211	-	-	-	-	-	-	NYSDEC No Further Action letter
OU1	RES212	-	-	-	-	-	-	NYSDEC No Further Action letter



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU1	RES213	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES214	-	-	-	-	-	-	No additional sampling
OU1	RES215	Category B	3 SB	Around SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At SS003	2 in bgs	0-2"	TAL Metals (excluding COPCs), Mercury and SVOCs	-
		Category E	1 SS	At SS009	2 in bgs	0-2"	SVOCs	-
		Category E	1 SS	At SS010	2 in bgs	0-2"	SVOCs	-
		Category B	2 SS	Northwest and northeast of SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	Southwest of SS004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	Northwest of SS006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	East of SS007	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	Northwest and southwest of SS012	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU2	RES216	Category B	2 SS	East and Southwest of SS013	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU2	RES216	Category B	1 SB	West of SB003	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Along southeast property line	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU1	RES218	Category B	1 SB	North of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of SB005	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	Northwest of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category C	1 SB	Between SB001 and SB005	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU1	ROW	Category B	1 SB	North of ROWSB045	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of ROWSB025 and south of RES218SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES219	-	-	-	-	-	-	Property included in Expedited Sampling Plan
OU1	RES220	Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	No soil borings on property to date
OU1	RES221	Category B	1 SB	East of SB002	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	3 SB	North, South and West of SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around SB004	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of SB004	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB006	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
OU2	RES222	-	-	Sample location SS003	-	-	-	No action per NYSDEC for barium at 147 (379J) mg/kg
		Category B	1 SB	In backyard	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	2 SB	North and East of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of SB001	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category C	1 SB	Northeast corner of property	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU2	ROW	Category B	1 SB	South of RES222SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	North and East of SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU2	RES223	Category C	3 SB	Back yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB002	2 ft bgs	0-2" and 2"-24"	TAL Metals (excluding COPCs), mercury and SVOCs	Cover System Sample
OU2	ROW	Category B	1 SB	South of RES223SB001	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU1	RES229	Category C	2 SB	Backyard along property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SB001	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SS	At SB002	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category E	1 SB	At SB003	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		Category E	1 SB	At SB004	2 ft bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
						2"-24"	SVOCs	
		Category E	1 SS	At SB005	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA001	Category E	1 SS	At SB006	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	4 SB	Around 001-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 001-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA002	-	-	Sample location 001-B2	-	-	-	No action per NYSDEC for arsenic of 19.9 mg/Kg (15-16')
		-	-	-	-	-	-	No additional sampling



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU5	EA003	Category B	4 SB	Around 003-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around 003-B6	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	2 SB	Backyard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA004	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA005	Category B	3 SB	Around 005-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 005-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around 005-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	ROW	Category B	1 SS	South of 0705-S1 in ROW	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA006	Category B	4 SS	Around 006-S4	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 006-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 006-B2	-	-	-	No action per NYSDEC for arsenic at 18.1 mg/kg and manganese at 3,010 mg/kg (15-16')
OU5	EA007	Category B	1 SB	Between 007-B1 and 007-B4	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category C	1 SB	Front yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA008	Category B	4 SB	Around 008-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	East and West of 008-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	North, East and South of 008-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 008-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 008-B3	-	-	-	No action per NYSDEC for manganese at 4320 mg/Kg (15-16')
		-	-	-	-	-	-	-
OU5	EA009	Category B	2 SB	North and south of 009-B1	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	South of 009-B4	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SS	South and west of 009-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Near 009-S4	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU5	ROW	Category B	1 SB	East of 009-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA010	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA011	Category B	4 SS	Around 011-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 011-S1	-	-	-	No action per NYSDEC for arsenic at 17.9 mg/Kg
OU5	EA012	Category B	4 SB	Around 012-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 012-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA013	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA014	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA015	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA016	Category B	3 SB	Around 016-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around 016-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Along eastern property line	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category C	2 SB	Along southern property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA017	Category B	1 SB	Along southern property line	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	3 SB	Around 017-B2	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southwest of 017-B2	2 ft bgs	0-2" and 2"-24"	Tal Metals, Mercury and SVOCs	-
		Category B	1 SB	Northeast of 017-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Southeast of 017-B1	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	3 SB	North, West and South of 017-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At 017-B4	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category B	2 SB	East property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU5	ROW	Category B	2 SB	Northwest and Southwest of 017-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA018	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA019	-	-	-	-	-	-	No additional sampling
OU5	EA020	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA021	-	-	-	-	-	-	No additional sampling
OU5	EA022	Category B	3 SB	Around 022-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around 022-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA023	Category B	4 SB	Around 023-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA024	-	-	-	-	-	-	No additional sampling
OU5	EA025	-	-	-	-	-	-	NYSDEC No Further Action letter



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU5	EA026	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA027	-	-	-	-	-	-	No additional sampling
OU5	EA028	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA029	Category B	1 SB	Along southern property line	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property Soil boring to limit area of institutional control
		Category B	3 SB	Around 029-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	South of 029-B1	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southwest of 029-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	East and Southeast of 029-B3	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SS	North and West of 029-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	North and Southeast 029-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category A	2 SS	To be determined in the field (backyard)	2 in bgs	0-2"	COPCs	Only 2 surface soil samples collected to date.
OU5	EA030	-	-	Sample locations 030-B1 and 030-B4	-	-	-	No Soil Boring in backyard to date.
		Category C	1 SB	South of 030-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	No action per NYSDEC for exceedances at depths greater than 2 ft bgs
OU5	EA031	-	-	-	-	-	-	Cover System Sample
OU5	EA032	-	-	-	-	-	-	No additional sampling
		Category B	2 SB	Northeast and Southwest of 032-B5	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	2 SB	West and South of 032-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	North, West and south of 032-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SB	At 032-B3	2 ft bgs	0-2" and 2"-24"	SVOCs	-
		Category B	3 SB	Around 032-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA034	Category B	2 SS	North and East of 032-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	-	-	-	-	No additional sampling
OU5	EA035	-	-	Sample location 034-S4	-	-	-	No action per NYSDEC for SVOC at ND
		Category B	3 SB	North, East and Southwest of 035-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 035-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	ROW	Category C	3 SB	North, East and Southeast of 035-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA036	Category B	1 SB	North of ROW-B6	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA037	Category B	1 SB	Along west property boundary	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	-
OU5	EA038	-	-	-	-	-	-	Based on sample results from adjacent property
OU5	EA039	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA040	-	-	-	-	-	-	NYSDEC No Further Action letter
		-	-	Sample location EA039-G2	-	-	-	No additional sampling
OU5	EA041	-	-	-	-	-	-	No action per NYSDEC for arsenic at 37J mg/Kg
OU5	EA042	-	-	-	-	-	-	NYSDEC No Further Action letter
		Category B	4 SB	Around 041-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around 041-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA043	Category B	3 SS	Around 042-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 042-B1	-	-	-	No action per NYSDEC for manganese at 2,190 mg/kg (15-16')
OU5	EA044	-	-	-	-	-	-	-
OU5	EA045	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA046	Category B	4 SB	Around 045-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SS	Along southern property boundary	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU5	EA047	Category B	3 SB	Around 046-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA048	Category B	4 SS	Around 047-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 047-S3	-	-	-	-
OU5	EA049	-	-	Sample location 047-S3	-	-	-	No action per NYSDEC for arsenic at 16.9 mg/Kg
OU5	EA050	Category B	4 SB	Around 048-B2	15 ft bgs	2'-4'	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU5	EA051	Category D	1 SB	Near backyard shed	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Required by NYSDEC
		-	-	Sample location 049-G2	-	-	-	No action per NYSDEC for arsenic at 16.3 mg/Kg
OU5	EA052	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2" , 2"-24" and native	TAL Metals and Mercury	Property not sampled to date



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU5	EA051	Category B	1 SB	Between 051-B1 and 051-B2	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category C	1 SB	Northwest corner of house	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	1 SB	South of 051-B1	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SS	East of 051-S4	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of 051-S4	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property Soil boring for endpoint and to limit area of institutional control
OU5	EA052	Category B	3 SB	Around 052-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	South of 052-B3	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Along southern property line	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	1 SB	East of 052-B4	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category C	1 SB	South of 052-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		-	-	Sample locations 052-B1 and 052-B2	-	-	-	No action per NYSDEC for arsenic and manganese at 15-16'
OU5	EA053	Category B	4 SB	Around 053-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SS	East and west of 053-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA054	Category B	2 SB	East and West of 054-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	East and West of 054-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	South of 054-B3	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southeast property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU5	EA055	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA056	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA057	-	-	-	-	-	-	No additional sampling
OU5	EA058	Category B	3 SS	Around 058-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 058-B2	-	-	-	No action per NYSDEC for arsenic at 27.6 mg/kg (8-10')
OU5	ROW	Category B	1 SS	South of 058-S1 in ROW	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA059	Category C	7 SB	Across Property	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA060	Category B	4 SB	Around 060-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SB	Around 060-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	4 SS	Around 060-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA061	Category B	4 SB	Around 061-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		-	-	Sample location 061-S2	-	-	-	No action per NYSDEC for SVOCs
OU5	EA062	Category B	2 SB	North and west of 062-B4	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Along the southern property line	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property Soil boring for endpoint and to limit area of institutional control
OU5	EA063	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA064	Category B	4 SB	Around 064-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 064-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA065	Category B	3 SB	Around 065-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Backyard, North and South	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category C	1 SB	Along southern property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	4 SB	Around 065-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA066	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA067	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA068	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA069	Category C	2 SB	Front yard and side yard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	1 SB	West of 069-B4	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	East of 069-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	Southwest and southeast of 069-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of 069-B6	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
OU5	EA070	Category B	1 SS	South of 070-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	South and Southwest of 070-B2	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	West of 070-B3	15 ft bgs	0-2" , 2"-24" and native	TAL Metals, Mercury and SVOCs	-
OU5	ROW	Category B	1 SB	South of ROW-B6	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU5	EA071	Category B	4 SB	Around 071-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	West and east 071-B2	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
OU5	EA072	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU5	EA073	Category B	4 SB	Around 073-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 073-S4	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SB	Around 073-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 073-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Along eastern property boundary	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
		Category B	1 SS	South of 073-S1 in ROW	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA074	Category B	2 SB	West and North of 074-B4	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	2 SB	Northwest and East of 074-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA075	Category B	3 SB	Around 075-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 075-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA076	Category B	1 SB	Northeast corner of house	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of 076-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	Center of backyard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
OU5	EA077	Category B	1 SB	East of 077-B1	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	East of 077-B2	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	Southeast of 077-B2	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	4 SB	Around 077-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	3 SS	Around 077-S2	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA078	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA079	Category B	3 SB	Around 079-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At 079-B1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category E	1 SS	At 079-B3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	EA080	Category B	3 SS	Around 080-S3	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
OU5	ROW	Category B	2 SB	North and south of ROW-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA081	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA082	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA083	-	-	-	-	-	-	No additional sampling
OU5	EA084	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA085	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA086	-	-	-	-	-	-	No additional sampling
OU5	EA087	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU5	EA088	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date
OU5	EA089	-	-	-	-	-	-	No additional sampling
OU5	EA090	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA091	Category B	4 SB	Around 091-B2	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA092	Category B	2 SB	South and West of 092-B3	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring for endpoint and to limit area of institutional control
		Category B	1 SB	East of 092-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category C	1 SB	North side of backyard	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	1 SB	Backyard (east of house)	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		-	-	Sample location 092-B2	-	-	-	No action per NYSDEC for mercury at 1.7 mg/Kg (3-4')
OU5	EA093	-	-	-	-	-	-	No additional sampling
OU5	EA094	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA095	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA096	-	-	-	-	-	-	No additional sampling
OU5	EA097	-	-	-	-	-	-	NYSDEC No Further Action letter



Table 4-4
Pre-Design Investigation Sample Summary
(continued)

OU	Property	Sample Type	Number/ Type	Location	Depth	Sample Interval(s)*	Analytics	Notes
OU5	EA098	Category C	1 SB	West of 098-B1	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	Cover System Sample
		Category B	4 SS	Around 098-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	North and East of 098-B1	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Soil boring to limit area of institutional control
		Category B	1 SB	North of 098-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of 098-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	North of 098-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	ROW	Category B	3 SB	East, West and South of 098-B3	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	South of 098-B4	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	East and South 098-B5	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
		Category B	2 SB	South and East of ROW-T7	2 ft bgs	0-2" and 2"-24"	TAL Metals, Mercury and SVOCs	-
OU5	EA099	Category B	2 SS	Around 099-S1	2 in bgs	0-2"	TAL Metals, Mercury and SVOCs	-
		Category B	1 SB	Along northern property boundary	15 ft bgs	0-2", 2"-24" and native	TAL Metals, Mercury and SVOCs	Based on sample results from adjacent property
OU5	EA100	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA101	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA102	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA103	-	-	-	-	-	-	No additional sampling
OU5	EA104	-	-	-	-	-	-	No additional sampling
		-	-	Sample location 104-S4	-	-	-	No action per NYSDEC for arsenic at 9.7J mg/Kg (16.1J mg/Kg)
OU5	EA105	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA106	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA107	-	-	-	-	-	-	No additional sampling
OU5	EA108	-	-	-	-	-	-	NYSDEC No Further Action letter
OU5	EA109	-	-	-	-	-	-	No additional sampling
OU5	EA110	Category A	4 SS	To be determined in the field	2 in bgs	0-2"	COPCs	Property not sampled to date
		Category A	2 SB	To be determined in the field	15 ft bgs	0-2", 2"-24" and native	TAL Metals and Mercury	Property not sampled to date

Notes:
COPCs - Constituents of Potential Concern (i.e., arsenic, cadmium and lead)
ROW - City of Corning right-of-way
SB - soil boring
SS - surface soil
SVOC - semi-volatile organic compound
TAL - Target Analyte List
ND - Not detected



5. PROJECT MANAGEMENT

5.1 SCHEDULE

The activities described in this PDI Work Plan are expected to be performed following the effective date of the Order between Corning Incorporated and NYSDEC and upon receipt of consent to access from property owners. The project schedule for the PDI activities is provided as Figure 5-1. It should be noted that the planned activities are not independent and the schedule and approach may be adjusted accordingly as work is completed within OU1, OU2 and OU5. The schedule will be updated every other week and submitted to NYSDEC and NYSDOH. Furthermore, this work schedule is predicated on obtaining written access consent from property owners in a timely fashion.

5.2 DOCUMENTATION

5.2.1 Field Logs

Essential project information pertinent to field activities, including sampling, will be recorded in bound field logbooks with consecutively numbered pages and/or field data record forms specific to a given activity. Entries into the logbook will typically contain information such as:

- Date and time of logbook entry
- Names of all team members present
- Weather conditions
- Field observations
- Log and summary of daily activities and significant events
- Description of sample and sampling location
- Date and time of sample collection
- Collector's sample identification number(s) and/or name
- Name and affiliation of personnel or visitors
- Decontamination activities
- Description of any problem encountered and problem resolution



Entries will be made in ink with no erasures. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed, and dated.

5.2.2 Photo Log

A project photo log will be prepared and maintained throughout the pre-design investigation activities to provide photo documentation of field activities. In particular, photos of the soil boring cores will be taken and logged.

5.2.3 Field Reports

WESTON will prepare brief daily work activity reports concisely summarizing the work performed each day. At the completion of the work, all record documents will be provided to Corning Incorporated. During the execution of the work described in this PDI Work Plan, WESTON will provide NYSDEC and NYSDOH verbal updates of the field activities periodically, and electronic copies of weekly work activity reports, including select supporting photographs. WESTON will record all ambient air monitoring data in the field logbook or designated field sheets and communicate the results of the air monitoring to the NYSDEC and NYSDOH on a scheduled basis (i.e. daily for levels which require actions, weekly for routine monitoring data). At the completion of the field work and data validation activities described in this PDI Work Plan, WESTON will provided monthly work activity reports to NYSDEC and NYSDOH.

5.2.4 Data Management

Laboratory analytical data will be managed by WESTON in an electronic database and will be uploaded in an electronic data deliverable (EDD) format.

WESTON will review laboratory data deliverable packages for completeness, adherence to holding times, comparison with chain-of-custody, etc. WESTON will perform data validation and prepare a DUSR. The data review/validation activities are described in the QAPP provided in Appendix C.

5.2.5 Reporting

Upon receipt of validated data as it is generated throughout the project, Corning Incorporated will supply such validated data to NYSDEC in DUSR. The DUSRs will include tables summarizing sample analytical results as well as tables summarizing QA/QC sample results.



NYSDEC will review the data and Corning Incorporated's transmittal letters before they are provided to individual property owners.

5.3 HEALTH AND SAFETY PLAN

The health and safety of field workers, clients, visitors, and the community are of utmost importance. For the field work, it is planned that workers will be in Level D personal protection (i.e., coveralls or work clothes, work boots, safety glasses, and hard hats). All field activities will be conducted in accordance with the HASP and CAMP provided in Appendix A and Appendix B, respectively.

5.4 STUDY AREA CONTROLS

A temporary field office and equipment storage area is located in a NYSDEC-approved staging area near the Study Area. This temporary field office area is surrounded by temporary fencing for security and the access gate is closed and locked when not in use.

The temporary field office area presently consists of an office trailer for document and sample preparation, and staging area for field equipment. Electricity is currently supplied to the mobile office via a power drop.

5.5 COMMUNITY RELATIONS

A supplemental Citizen Participation Plan (CPP) for the Study Area has been prepared in accordance with DER-10 requirements and submitted to the NYSDEC. The CPP describes the community relations components to be followed during the implementation of this PDI Work Plan and other Study Area related activities.



SECTION 5

FIGURE

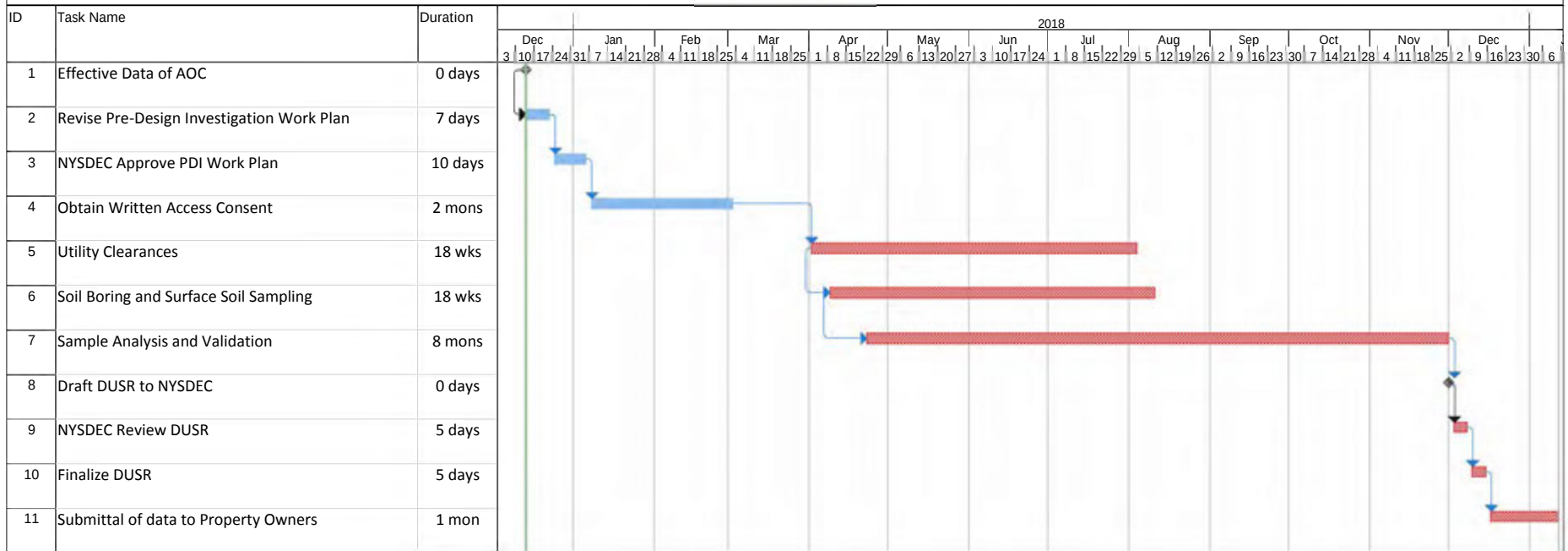


Figure 5-1
Pre-Design Work Plan Schedule
Study Area, Corning, NY

Task Split Milestone Critical Path



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APPENDIX A

HEALTH AND SAFETY PLAN (HASP)

The final Health and Safety Plan will be maintained at the Study Area during field activities.



DRAFT

HEALTH AND SAFETY PLAN (HASP)

Office: West Chester, PA
Project Name: Study Area Bounded by the Chemung River; Post Creek; I-86; the Guthrie Medical Center, the City of Corning Fire Department and Centerway.
Client: Corning Incorporated
Work Location: Corning, NY
WO#: 02005.056.002.0001





BEHAVIOR-BASED SAFETY (BBS) – Pledge

I Accept and Understand 100% Safe Work Is an Achievable Goal

- ★ I will work to develop strong connections and team with my co-workers to establish a culture of working safely 100% of the time.
- ★ I will actively care about all Weston employees, our families, team contractors and clients.
- ★ I will help to keep our projects safe and will meet and exceed compliance requirements.
- ★ I will understand and comply with the Health and Safety Plan, Accident Prevention Plan, and Environmental Compliance Plan for each field project. They guide my actions.
- ★ I will stop any work that presents an imminent hazard to people or the environment or is not adequately addressed in the Health and Safety Plan, Accident Prevention Plan, or Environmental Compliance Plan.
- ★ I will identify changing conditions to address safety implications. No surprises!
- ★ I will identify unsafe working conditions and be proactive in correcting them.
- ★ I will coach and mentor and will accept coaching from others to encourage safe work behaviors.
- ★ I am empowered to share lessons-learned and foster continuous improvement.

I will Learn where I can get Assistance

- ★ I will develop high quality relationships with my Division Environmental, Health, and Safety (EHS) Manager; Profit Center Safety Officer; and Field Safety Officer.
- ★ I will learn how and when to contact our Environmental Advisors.
- ★ I will get to know our Corporate EHS staff and become familiar with the Corporate EHS Portal Site.

I will Report All Incidents

- ★ If a safety incident occurs, even if there is no injury or damage but there could have been, I will report the incident immediately.
- ★ I will conduct safety reviews of all incidents with my supervisor, if requested. The review will focus on cause and lessons-learned so that we can be proactive in preventing it from happening again.



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ATTACHMENTS

ATTACHMENT A	Chemical Contaminants Data Sheets
ATTACHMENT B	Safety Data Sheets
ATTACHMENT C	Safety Procedures/Field Operating Procedures (FLD Ops)
ATTACHMENT D	Hazard Communication Program
ATTACHMENT E	Air Sampling Data Sheets
ATTACHMENT F	Incident Reporting
ATTACHMENT G	Traffic Control Plan
ATTACHMENT H	Environmental Health & Safety Inspection Checklist
ATTACHMENT I	Community Air Monitoring Plan



1. PERSONNEL ON SITE INFORMATION



1.1 WESTON REPRESENTATIVES

Organization/Branch	Name/Title	Address	Telephone
National Accounts	John Sontag/Project Manager	1400 Weston Way West Chester, PA 19380	610-701-3679
National Accounts	Rachel McLoughlin/Senior Project Scientist	1400 Weston Way West Chester, PA 19380	610-701-3428
National Accounts	Audra Balson / Senior Geoscientist	1400 Weston Way West Chester, PA 19380	610-701-3037
National Accounts	Devon Hollenden/Assistant Geoscientist 2	1400 Weston Way West Chester, PA 19380	610-701-3091

Roles and Responsibilities:

Manage and implement Study Area sampling program.

1.2 WESTON SUBCONTRACTORS

Organization/Branch	Name/Title	Address	Telephone
Nothnagle Drilling, Inc.	Name: Stephen A. DiLaura Title: Vice President / Geologist	Street: 1821 Scottsville-Mumford Road City: Scottsville State, Zip: New York, 14586	585-538-2328
New York Leak Detection, Inc.	Name: Laurie Bobbette Title: Office Manager	Street: PO Box 269 - 4538 North St. City: Jamesville State, Zip: New York, 13078	315-469-4601
	Name: Title:	Street: City: State, Zip:	

Roles and Responsibilities:

Nothnagle drilling is the drilling subcontractor responsible for all geoprobe related-activities

New York Leak Detection, Inc. is the third-party utility locator

SITE-SPECIFIC HEALTH AND SAFETY PERSONNEL

The Site Field Safety Officer (FSO) for activities to be conducted at this site is: Rachel McLoughlin

The Site Manager has ultimate responsibility for ensuring that the provisions of this Site HASP are adequate and implemented in the field.

Changing field conditions may require decisions to be made concerning adequate protection programs. Therefore, the personnel assigned as FSOs must be experienced and meet the additional training requirements specified by OSHA in 29 CFR 1910.120.

Qualifications:

40-hour OSHA HAZWOPER certification; annual 8-hour OSHA HAZWOPER refresher certification; 8-hour Site Health and Safety Officer certification; current Adult First Aid and CPR certification; familiarity with jobs of similar scope.

Designated alternates include: Devon Hollenden, Audra Balson



1.3 SITE PERSONNEL AND CERTIFICATION STATUS

1.3.1 WESTON Employee Certification

Name: John Sontag Title: Project Manager Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Rachel McLoughlin Title: Senior Project Scientist Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)
Name: Devon Hollenden Title: Assistant Geoscientist 2 Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Sarah Bonya Title: Assistant Project Scientist Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)
Name: Drew Eberts Title: Assistant Project Scientist Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Audra Balson, P.G. Title: Senior Geoscientist Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)
Name: Greg Flasinski Title: Technician 6 Task(s): All Certification Level or Description: ☒ Medical Current ☒ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)
Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)

TRAINING CURRENT - Training: All personnel, including visitors, entering the exclusion or contamination reduction zones must have certifications of completion of training in accordance with OSHA 29 CFR 1910, 29 CFR 1926, or 29 CFR 1910.120.

FIT TEST CURRENT - Respirator Fit Testing: All persons, including visitors, entering any area requiring the use or potential use of any tight-fitting respirator must have had, as a minimum, a qualitative fit test, administered in accordance with OSHA 29 CFR 1910.134 or ANSI, within the last 12 months. If site conditions require the use of a full-face, tight-fitting, air-purifying respirator for protection from asbestos or lead, employees must have had a quantitative fit test, administered according to OSHA 29 CFR 1910.1001 or .1025 or 29 CFR 1926.1101 or .62, within the last 12 months.

MEDICAL CURRENT - Medical Monitoring Requirements: All personnel, including visitors, entering the exclusion or contamination reduction zones must be certified as medically fit to work and able to wear a respirator, if appropriate, in accordance with 29 CFR 1910 or 29 CFR 1926 (substance-specific), or 29 CFR 1910.120 (HAZWOPER).

The Site Field Safety Officer is responsible for verifying all certifications and fit tests.



SITE PERSONNEL AND CERTIFICATION STATUS		
1.3.2 Subcontractor's Health and Safety Program Evaluation		
Name of Subcontractor: Nothnagle Drilling Address: 1821 Scottsville-Mumford Road Scottsville, NY 14586		
Activities To Be Conducted by Subcontractor: Drilling/geoprobe related-activities		
Evaluation Criteria		
Medical Program meets OSHA/WESTON criteria ☐ Acceptable ☐ Unacceptable Comments:	Personal Protective Equipment available ☐ Acceptable ☐ Unacceptable Comments:	On-site monitoring equipment available, calibrated, and operated properly ☐ Acceptable ☐ Unacceptable Comments:
Safe Working Procedures clearly specified ☐ Acceptable ☐ Unacceptable Comments:	Training meets OSHA/WESTON criteria ☐ Acceptable ☐ Unacceptable Comments:	Emergency Procedures ☐ Acceptable ☐ Unacceptable Comments:
Decontamination Procedures ☐ Acceptable ☐ Unacceptable Comments:	General Health and Safety Program evaluation ☐ Acceptable ☐ Unacceptable Comments:	Additional comments: ☐ Subcontractor has agreed to and will conform to the WESTON HASP for this project. ☒ Subcontractor will work under its own HASP
Evaluation Conducted by: N/A Evaluation Source (SubTrack, etc.):		Date:
Subcontractor		
Certifications for all subcontractor personnel will be added to the HASP prior to beginning work.		
Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	
Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	Name: Title: Task(s): Certification Level or Description: ☐ Medical Current ☐ Training Current ☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)	



SITE PERSONNEL AND CERTIFICATION STATUS

1.3.3 Subcontractor's Health and Safety Program Evaluation

Name of Subcontractor: New York Leak Detection

Address: PO Box 269 - 4538 North St.
Jamesville, NY 13078

Activities To Be Conducted by Subcontractor: Utility Clearance and Markout

Evaluation Criteria

Medical Program meets OSHA/WESTON criteria

- ☐ Acceptable
☐ Unacceptable

Comments:

Personal Protective Equipment available

- ☐ Acceptable
☐ Unacceptable

Comments:

On-site monitoring equipment available, calibrated, and operated properly

- ☐ Acceptable
☐ Unacceptable

Comments:

Safe Working Procedures clearly specified

- ☐ Acceptable
☐ Unacceptable

Comments:

Training meets OSHA/WESTON criteria

- ☐ Acceptable
☐ Unacceptable

Comments:

Emergency Procedures

- ☐ Acceptable
☐ Unacceptable

Comments:

Decontamination Procedures

- ☐ Acceptable
☐ Unacceptable

Comments:

General Health and Safety Program evaluation

- ☐ Acceptable
☐ Unacceptable

Comments:

Additional comments:

- ☐ Subcontractor has agreed to and will conform to the WESTON HASP for this project.
☒ Subcontractor will work under its own HASP

Evaluation Conducted by:

Date:

Evaluation Source (SubTrack, etc.):

Subcontractor

Certifications for all subcontractor personnel will be added to the HASP prior to beginning work.

Name:

Title:

Task(s):

Certification Level or Description:

- ☐ Medical Current ☐ Training Current
☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)

Name:

Title:

Task(s):

Certification Level or Description:

- ☐ Medical Current ☐ Training Current
☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)

Name:

Title:

Task(s):

Certification Level or Description:

- ☐ Medical Current ☐ Training Current
☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)

Name:

Title:

Task(s):

Certification Level or Description:

- ☐ Medical Current ☐ Training Current
☐ Fit Test Current (Qual.) ☐ Fit Test Current (Quant.)



2. HEALTH AND SAFETY EVALUATION



2.1 HEALTH AND SAFETY EVALUATION

2.1.1 Task Hazard Assessment

Background Review: ☒ Complete ☐ Partial If partial why? **N/A**

Activities Covered Under This Plan:

No.	Task/Subtask	Description	Schedule
1	Soil sampling	Collect soil boring and surface soil samples. Provide oversight of drilling subcontractor.	Ongoing
2	Groundwater Monitoring Well Installation	Provide oversight of drilling subcontractor during installation of groundwater monitoring wells.	TBD
3	Groundwater Sampling	Collect groundwater samples from site monitoring wells.	TBD
4	Test Pits/Excavation	Excavation and backfill of test pits/surface soil.	TBD
5	Remediation	Oversight of subcontractor performing excavation of soils and/or layers of fill material containing ash, brick and/or glass up to 2 feet bgs.	TBD

Types of Hazards:

Numbers refer to one of the following hazard evaluation forms. Complete hazard evaluation forms for each appropriate hazard class.

Physiochemical 1

- ☐ Flammable
☐ Explosive
☐ Corrosive
☐ Reactive
☐ O₂ Rich
☐ O₂ Deficient

Chemically Toxic 1

- ☒ Inhalation ☒ Carcinogen
☒ Ingestion ☐ Mutagen
☒ Contact ☐ Teratogen
☐ Absorption
☐ OSHA 1910.1000 Substance (Air Contaminants)
☒ OSHA Specific Hazard Substance Standard (Refer to following page for listing)

Radiation 3

- Ionizing:
☐ Internal exposure
☐ External exposure

 Non-ionizing:
☒ UV ☐ IR
☐ RF ☐ MicroW
☐ Laser

Biological 2

- ☐ Etiological Agent
☒ Other (plant, insect, animal)

Physical Hazards 4

- ☒ Characterization Activities

Source/Location of Contaminants and Hazardous Substances:

Directly Related to Tasks

- ☐ Air
☐ Other Surface
☒ Groundwater
☒ Soil
☐ Surface Water
☐ Sanitary Wastewater
☐ Process Wastewater
☐ Other _____

Indirectly Related to Tasks — Nearby Process(es) That Could Affect Team Members:

- ☐ WESTON Work Location
☒ Nearby Non-Client Facility

Describe: **See Comments**

- ☐ Have activities (task[s]) been coordinated with facility?

Comments:

Work is being performed on City of Corning and privately owned residential property and requires a signed access agreement by the property owner prior to any activities



HEALTH AND SAFETY EVALUATION

2.1.2 Chemical Hazards of Concern

☒ N/A

Chemical Contaminants of Concern

Attach data sheets from an acceptable source such as NIOSH pocket guide, condensed chemical dictionary, ACGIH TLV booklet, Hazardous Substances Data base (HSDB), etc. List chemicals and concentrations below and locate data sheets in Attachment A of this HASP.

☐ N/A

Identify hazardous materials used or on-site and attach Safety Data Sheets (SDSs) for all reagent type chemicals, solutions, or other identified materials that in normal use in performing tasks related to this project could produce hazardous substances. Ensure that all subcontractors and other parties working nearby are informed of the presence of these chemicals and the location of the SDSs. Obtain from subcontractors and other parties, lists of the hazardous materials they use or have on-site and identify location of the SDSs here. List chemicals and quantities below and locate SDSs in Attachment B of this HASP.

Chemical Name	Concentration (mg/Kg)	Chemical Name	Quantity
Arsenic	*See Table on next page		
Cadmium			
Lead			

OSHA-SPECIFIC HAZARDOUS SUBSTANCES

☐ 1910.1001 Asbestos	☐ 1910.1002 Coal tar pitch volatiles	☐ 1910.1003 4-Nitrobiphenyl, etc.	☐ 1910.1004 alpha-Naphthylamine
☐ 1910.1005 [Reserved]	☐ 1910.1006 Methyl chloromethyl ether	☐ 1910.1007 3,3'-Dichlorobenzidine (and its salts)	☐ 1910.1008 bis-Chloromethyl ether
☐ 1910.1009 beta-Naphthylamine	☐ 1910.1010 Benzidine	☐ 1910.1011 4-Aminodiphenyl	☐ 1910.1012 Ethyleneimine
☐ 1910.1013 beta-Propiolactone	☐ 1910.1014 2-Acetylaminofluorene	☐ 1910.1015 4-Dimethylaminoazobenzene	☐ 1910.1016 N-Nitrosodimethylamine
☐ 1910.1017 Vinyl chloride	☒ 1910.1018 Inorganic arsenic	☒ 1910.1025 Lead (Att. FLD# 46)	☐ 1910.1026 Chromium VI (att. FLD 53)
☒ 1910.1027 Cadmium (Att. 50 FLD)	☐ 1910.1028 Benzene (Att. FLD# 54 or 61)	☐ 1910.1029 Coke oven emissions	☐ 1910.1043 Cotton dust
☐ 1910.1044 1,2-Dibromo-3-chloropropane	☐ 1910.1045 Acrylonitrile	☐ 1910.1047 Ethylene oxide	☐ 1910.1048 Formaldehyde
☐ 1910.1050 Methylenedianiline	☐ 1910.1051 1,3 Butadiene	☐ 1910.1052 Methylene chloride	☐ 1926.60 Methylenedianiline
☒ 1926.62 Lead	☐ 1926.1101 Asbestos (Att. FLD 52)	☒ 1926.1127 Cadmium	



	Screening Levels¹	Minimum Analytical Result (mg/Kg)	Maximum Analytical Result (mg/Kg)	Average Analytical Result (mg/Kg)	Total # Samples	Total # Samples above the Screening Level	Percentage of Analytical Results < SCO
Arsenic, Total	16 / 16	0.50 U	1,280	13.5	4,585	455	90.08%
Cadmium, Total	2.5 / 4.3	0.098 U	21,000	6.4	4,585	212	95.38%
Lead, Total	63 / 400	0.30 U	28,600	167	4,585	1,498	67.33%

¹ Screening levels are displayed Residential SCOs / Restricted-Residential SCOs.

NYSDEC = New York State Department of Environmental Conservation

Residential SCO = NYSDEC Subpart 375-6 Remedial Program Residential Soil Cleanup Objective.

Restricted-Residential SCO = NYSDEC Subpart 375-6 Remedial Program Restricted-Residential Soil Cleanup Objective.

Samples from residential areas are screened against Residential SCOs while samples from Rights-of-Way areas are screened against Restricted-Residential SCOs.

Data in this table collected in the residential areas of the Study Area from June 2014 – November 2015.



HEALTH AND SAFETY EVALUATION

2.1.3 Biological Hazards of Concern

☒ **Poisonous Plants** (FLD 43-D)

Location/Task No(s) **All**

Source: ☐ Known ☒ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☒ Contact ☐ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☒ No
 Immunization required: ☐ Yes ☒ No

☒ **Insects** (FLD 43-B)

Location/Task No(s) **All**

Source: ☐ Known ☒ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☒ Contact ☒ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☒ No
 Immunization required: ☐ Yes ☒ No

☒ **Snakes, Reptiles** (FLD 43-A)

Location/Task No(s) **All**

Source: ☐ Known ☒ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☒ Contact ☒ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☒ No
 Immunization required: ☐ Yes ☒ No

☒ **Animals** (FLD 43-A)

Location/Task No(s) **All**

Source: ☐ Known ☒ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☒ Contact ☒ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☒ No
 Immunization required: ☐ Yes ☒ No

FLD 43 — WESTON Biohazard Field Operating Procedures: Att. OP ☐

☐ **Sewage**

Location/Task No.(s):

Source: ☐ Known ☐ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☐ Contact ☐ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☐ No
 Immunization required: ☐ Yes ☐ No

Tetanus Vaccination within Past 10 yrs: ☐ Yes ☐ No

☐ **Etiologic Agents** (FLD -C)(List)

Location/Task No.(s):

Source: ☐ Known ☐ Suspect
 Route of Exposure: ☐ Inhalation ☐ Ingestion
☐ Contact ☐ Direct Penetration

Team Member(s) Allergic: ☐ Yes ☐ No
 Immunization required: ☐ Yes ☐ No

FLD 43-C — Mold and Fungus. Att. OP ☐

FLD 44 — WESTON Bloodborne Pathogens Exposure Control Plan – First Aid Procedures: Att. OP ☒

FLD 45 — WESTON Bloodborne Pathogens Exposure Control Plan – Working with Infectious Waste: Att. OP ☐



HEALTH AND SAFETY EVALUATION								
2.1.4 Radiation Hazards of Concern								
NONIONIZING RADIATION								
Task No.	Type of Nonionizing Radiation	Source On-Site	TLV/PEL	Wavelength Range	Control Measures	Monitoring Instrument		
1	Ultraviolet	Solar			Appropriate clothing/sunscreen	None		
	Infrared							
	Radio Frequency							
	Microwave							
	Laser							
IONIZING RADIATION								
Task No.	Radionuclide	Major Radiations	Radioactive Half-Life (Years)	DAC ($\mu\text{Ci}/\text{mL}$)			Surface Contamination Limit	Monitoring Instrument
				D	W	Y		



HEALTH AND SAFETY EVALUATION

2.1.5 Physical Hazards of Concern

Physical Hazard Condition	Physical Hazard	Attach OP	WESTON OP Titles
Loud noise	Hearing loss/disruption of communication	☒	Section 7.0 - ECH&S Program Manual Occupational Noise & HC Program
Inclement weather	Rain/humidity/cold/ice/snow/lightning	☒	FLD02 - Inclement Weather
Steam heat stress	Burns/displaced oxygen/wet working surfaces	☐	FLD03 - Hot Process - Steam
Heat stress	Burns/hot surfaces/low pressure steam	☐	FLD04 - Hot Process - LT3
Ambient heat stress	Heat rash/cramps/exhaustion/heat stroke	☒	FLD05 - Heat Stress Prevention/Monitoring
Cold stress	Hypothermia/frostbite	☒	FLD06 - Cold Stress
Cold/wet	Trench/paddy/immersion foot/edema	☒	FLD02 - Inclement Weather
Confined spaces	Falls/burns/drowning/engulfment/electrocution	☐	FLD08 - Confined Space Entry
Industrial Trucks	Fork Lift Truck Safety	☐	FLD09 - Powered Industrial Trucks
Improper lifting	Back strain/abdomen/arm/leg muscle/joint injury	☒	FLD10 - Manual Lifting/Handling Heavy Objects
Uneven surfaces	Vehicle accidents/slips/trips/falls	☒	FLD11 - Rough Terrain
Poor housekeeping	Slips/trips/falls/punctures/cuts/fires	☒	FLD12 - Housekeeping
Structural integrity	Crushing/overhead hazards/compromised floors	☐	FLD13 - Structural Integrity
Improper cylinder. handling	Mechanical injury/fire/explosion/suffocation	☐	FLD16 - Pressure Systems - Compressed Gases
Water hazards	Poor visibility/entanglement/drowning/cold stress	☐	FLD17 - Diving
Water hazards	Drowning/heat/cold stress/hypothermia/falls	☐	FLD18 - Operation and Use of Boats
Water hazards	Drowning/frostbite/hypothermia/falls/electrocution	☐	FLD19 - Working Over Water
Vehicle hazards	Struck by vehicle/collision	☒	FLD20 - Traffic
Explosions	Explosion/fire/thermal burns	☐	FLD21 - Explosives
Moving mechanical parts	Crushing/pinch points/overhead hazards/electrocution	☒	FLD22 - Earth Moving Equipment
Moving mech. parts	Overhead hazards/electrocution	☐	FLD23 - Cranes, Rigging, and Slings
Working at elevation	Overhead hazards/falls/electrocution	☐	FLD24 - Aerial Lifts/Man lifts
Working at elevation	Overhead hazards/falls/electrocution	☐	FLD25 - Working at Elevation
Working at elevation	Overhead hazards/falls/electrocution/slips	☐	FLD26 - Ladders
Working at elevation	Slips/trips/falls/overhead hazards	☐	FLD27 - Scaffolding
Trench cave-in	Crushing/falling/overhead hazards/suffocation	☒	FLD28 - Excavating/Trenching
Physiochemical	Explosions/fires from oxidizing, flam./corr. material	☐	FLD30 - Hazardous Materials Use/Storage
Physiochemical	Fire and explosion	☒	FLD31 - Fire Prevention/Response Plan Required
Physiochemical	Fire	☒	FLD32 - Fire Extinguishers Required
Structural integrity	Overhead/electrocution/slips/trips/falls/fire	☐	FLD33 - Demolition
Electrical	Electrocution/shock/thermal burns	☒	FLD34 - Utilities
Electrical	Electrocution/shock/thermal burns	☒	FLD35 - Electrical Safety
Burns/fires	Heat stress/fires/burns	☐	FLD36 - Welding/Cutting/Brazing/Radiography
Impact/thermal	Thermal burns/high pressure impaction/heat stress	☒	FLD37 - Pressure Washers/Sand Blasting
Impaction/electrical	Smashing body parts/pinching/cuts/electrocution	☒	FLD38 - Hand and Power Tools
Poor visibility	Slips/trips/falls	☐	FLD39 - Illumination
Fire/explosion	Burns/impaction	☐	FLD40 - Storage Tank Removal/Decommissioning
Communications	Disruption of communications	☐	FLD41 - Std. Hand/Emergency Signals
Energy/release	Unexpected release of energy	☐	FLD42 - Lockout/Tag-out
Biological Hazards	Biological Hazards at site	☐	FLD43 - Biological Hazards
Animals	Animals	☒	FLD43A - Animals
Insects	Stinging and Biting Insects	☒	FLD43B - Stinging and Biting Insects
Molds/Fungi	Molds and Fungi	☐	FLD43C - Molds and Fungi



2.1.5 Physical Hazards of Concern (Continued)

Physical Hazard Condition	Physical Hazard	Attach OP	WESTON OP Titles
Hazardous Plants	Hazardous Plants	☒	FLD43D - Hazardous Plants
Etiologic Agents	Etiologic Agents	☐	FLD43E - Etiologic Agents
Biological Hazards/BBP	Biological Hazards/BBP at site/First Aid Providers	☒	FLD44 - Biological Hazards – Bloodborne Pathogens Exposure Control Plan – First Aid Providers
Infectious Waste	Infectious Waste at site/BBP/ at site/Infectious Waste	☐	FLD45 – Biological Hazards – Bloodborne Pathogens Exposure Control Plan – Work With Infectious Waste
Lead Contaminated sites	Lead poisoning	☒	FLD46 - Control of Exposure to Lead
Puncture/cuts	Cuts/ dismemberment/gouges	☐	FLD47 - Clearing, Grubbing and Logging Operations
Government Inspector	Disruption of Operations	☐	FLD48 – Federal, State, Local Regulatory Agency Inspections
Unknown Chemicals	Exposure to hazardous materials/waste	☒	FLD49 – Safe Storage of Samples
Cadmium	Exposure Control	☒	FLD50 – Cadmium Exposure Control Plan
Process Safety Procedure	Safety Procedure	☒	FLD51 – Process Safety Procedure
Asbestos	Asbestos Exposure	☐	FLD52 – Asbestos Exposure Control Plan
Hexavalent Chromium	Exposure Control Plan	☐	FLD53 – Hexavalent Chromium Exposure Control Plan
Benzene	Exposure Control Plan	☐	FLD54 - <u>Benzene Exposure Control Plan</u>
Hydrofluoric acid	Working with HF	☐	FLD55 – Working with Hydrofluoric Acid
Moving drill rig parts	Crushing/pinch points/overhead hazards/electrocution	☒	FLD56 – Drilling Safety
Vehicles/driving	Accidents,/fatigue/cell phone use	☒	FLD 57 – Motor Vehicle Safety
Improper material handling	Back injury/crushing from load shifts/equipment/tools	☒	FLD 58 – Drum Handling Operations
COC decontamination	COCs/slip, trip, and falls/waste generation/environmental compliance/PPE	☒	FLD59 - Decontamination
Drilling hazards	Electrocution/overhead hazards/pinch points	☒	Environmental Remediation Drilling Safety Guideline - 2005
Fatigue	Long work hours	☒	FLD60 – Employee Duty Schedule
Benzene/Gasoline	Benzene exposure	☐	FLD61 – Gasoline Contaminant Exposure
Cardiac Arrest	Accident/Heart Attack	☐	FLD62 – 2009 Automatic External Defibrillator (AED) Program Guidelines
Ionizing Radiation	Ionizing Radiation	☐	FLD63 – Using Handheld X-Ray Fluorescence (XRF) Analyzers
Working Alone	Isolated Working Conditions	☐	FLD64 – Employees Working Alone
Respiratory Protection	Fugitive Dust	☐	FLD65 – Respiratory Protection –APR, SCBA, Air-Line

**** Field Operation Procedures will be maintained on-site as a separate document and are also available on the WESTON portal ****



3. SITE SECURITY



3.1 SITE SECURITY ASSESSMENT FORM

DESCRIPTION	
Site Name and Location: Study Area, Corning NY	Number of Employees and Subcontractors on Site: TBD
Type of Work: Surface soil and soil boring sampling activities, with the potential for groundwater sampling from wells and oversight of subcontractor excavation activities.	
Projected Start Date: 2014 (On-going)	Projected Completion Date: TBD
Are Chemicals Used or Stored That Meet DHS/CFATS Requirements? N/A http://www.dhs.gov/files/programs/gc_1185909570187.shtm	
If Yes, Attach Plan and DHS Approvals to HASP. http://www.dhs.gov/files/programs/gc_1169501486197.shtm	
SURROUNDING AREA (<i>urban/suburban/rural; residential/commercial/industrial; traffic volume, population density, etc</i>) Suburban, residential neighborhood and school property within the Study Area limits.	
THREAT INDICATORS (<i>apparent social, economic, political, ethnic, criminal, gang related, and other risk factors</i>) N/A	
COUNTERMEASURES (<i>Current and projected risk mitigation factors</i>)	
Security Systems (Reference Site Security Checklist): Field office is surrounded by a chain link fence and is locked when unoccupied.	
Security Procedures (Reference Site Security Checklist): Field office is surrounded by a chain link fence and is locked when unoccupied.	
Closest police station location and contact information: Corning Police Department – 607-962-0340 1 Center Way Corning, NY 14830	
Other relevant observations or information to factor into the Site Security Plan: N/A	
OVERALL SECURITY ASSESSMENT (<i>Submit "Medium" and "High" risk assessments to Corporate Security for review</i>)	
Risk Level: ☒ Low ☐ Medium ☐ High	Date: 2016
Site Safety Officer: Rachel McLoughlin	Division Safety Manager: Thomas Drew
USE ATTACHMENTS FOR ADDITIONAL COMMENTS, MAPS AND DIAGRAMS	



3.2 WESTON SITE SECURITY CHECKLIST

*To be used for completing the Site Security Assessment Form required on all WESTON projects.
Contact Corporate Security for guidance on any items that are "NEEDED" and "NOT IN PLACE".*

CONTROL MEASURES:	In-Place / Not In-Place	Needed / Not Needed
1. Fencing, lockable gates, no holes (enter details below):	☒ / ☐	☐ / ☐
a. Chain Link material	☒ / ☐	☐ / ☐
b. Other material (describe)	☐ / ☐	☐ / ☒
c. Height (in feet and inches) 6'0"	☐ / ☐	☐ / ☒
d. Top cover (e.g., razor wire)	☐ / ☐	☐ / ☒
e. Signage (e.g., No Trespassing)	☐ / ☐	☐ / ☒
2. Guard service:	☐ / ☒	☐ / ☒
a. During working hours?	☐ / ☐	☐ / ☐
b. During non-working hours?	☐ / ☐	☐ / ☐
c. As a stationary post?	☐ / ☐	☐ / ☐
d. As a roving patrol?	☐ / ☐	☐ / ☐
e. Do they have written instructions?	☐ / ☐	☐ / ☐
f. Do they have adequate training?	☐ / ☐	☐ / ☐
g. Do they have adequate supervision?	☐ / ☐	☐ / ☐
h. Do they have daily reports?	☐ / ☐	☐ / ☐
i. Do they have daily inspections?	☐ / ☐	☐ / ☐
3. ID badges displayed by:	☐ / ☒	☐ / ☒
a. Employees?	☐ / ☐	☐ / ☐
b. Contractors?	☐ / ☐	☐ / ☐
c. Visitors?	☐ / ☐	☐ / ☐
4. Log books for:	☐ / ☒	☐ / ☒
a. Employee sign-in?	☐ / ☐	☐ / ☐
b. Visitor sign-in?	☐ / ☐	☐ / ☐
c. Vehicle sign-in?	☐ / ☐	☐ / ☐
d. Incident reports?	☐ / ☐	☐ / ☐
e. Property removal?	☐ / ☐	☐ / ☐
f. Keys and access cards?	☐ / ☐	☐ / ☐
5. Electronics and hardware options (enter details below):	☐ / ☒	☐ / ☒
a. Access card readers	☐ / ☐	☐ / ☐
b. Adequate lighting	☐ / ☐	☐ / ☐
c. Closed circuit TV	☐ / ☐	☐ / ☐
d. Alarm system	☐ / ☐	☐ / ☐
e. Other (describe)	☐ / ☐	☐ / ☐
6. Procedures documented for:	☐ / ☒	☐ / ☒
a. Security training?	☐ / ☐	☐ / ☐
b. Security instructions?	☐ / ☐	☐ / ☐
c. Contingency plans?	☐ / ☐	☐ / ☐
d. Opening and closing protocols?	☐ / ☐	☐ / ☐
e. Other (describe)?	☐ / ☐	☐ / ☐
7. Law enforcement liaison documented for:	☐ / ☒	☐ / ☒
a. Municipal police?	☐ / ☐	☐ / ☐
b. County sheriff?	☐ / ☐	☐ / ☐
c. State police?	☐ / ☐	☐ / ☐
d. Federal agencies (specify)?	☐ / ☐	☐ / ☐



WESTON SITE SECURITY CHECKLIST (CONTINUED)

*To be used for completing the Site Security Assessment Form required on all WESTON projects.
Contact Corporate Security for guidance on any items that are "NEEDED" and "NOT IN PLACE".*

CHAIN OF COMMAND:	Name	24/7 Contact Information
a. Security Coordinator	John Sontag	610-701-3679 (office); (484) 678-6135 (cell)
b. Site Supervisor	Rachel McLoughlin	610-701-3428 (office); (484) 433-2377 (cell)
c. Project Manager	John Sontag	610-701-3679 (office); (484) 678-6135 (cell)

REMARKS (use this section and supplemental pages to comment on details, exceptions or additional observations):

Field office is located in Corning, NY and consists of a small fenced in property. Inside the fence is a gravel parking lot consisting of two field office trailers, a conex box, which stores primarily PPE and field supplies (i.e., trash bags, paper towels, nitrile gloves, shovels, etc.) and a drum staging area. The drum staging area for investigative derived waste is in secondary containment and has an additional fence and lock (it remains locked at all times except when drums are being accessed). The outer fence is a 6 ft tall chain link fence which is locked every night. Additionally all field office trailers and the conex box are locked at all times when no Weston personnel is at the field office.



4. TASK BY TASK ASSESSMENT



4.1 TASK-BY-TASK RISK ASSESSMENT

4.1.1 Task 1 Description

TASK 1: Soil Sampling: This consists of surface soil sampling and/or soil boring sampling. Surface soil samples will be collected using a hand-auger; subsurface samples will be collected using a Geoprobe rig. Air monitoring will be conducted continuously by field personnel using two pDR meters, one upwind of sampling activities and one downwind. If levels are exceeded, field personnel will stop work, re-evaluate site conditions, and proceed in accordance with the Community Air Monitoring Plan (CAMP), attached.

EQUIPMENT REQUIRED/USED

Scoops	Hard hat
Hand tools	Plastic/Aluminum Trays
Nitrile gloves	pDR (dust monitoring)
Safety Boots	Mini-Rae (soil screening)
Safety Glasses	Drill Rig (if required)
Hearing Protection (if required)	Hand Auger (if required)
High-visibility vest (if required)	

POTENTIAL HAZARDS/RISKS

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Sampling soil with the potential presence of metal constituents.

Physical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Work generally will occur at residential or school property, with some work in utility right-of-way.

Biological

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Potential for ticks, bees, snakes, vegetation and small animals.

RADIOLOGICAL

☐ Hazard Present Risk Level: ☐ H ☐ M ☐ L

What justifies risk level?

N/A

LEVELS OF PROTECTION/JUSTIFICATION

Level D - safety shoes, safety glasses, hard hat, high-visibility vest (if required).

SAFETY PROCEDURES REQUIRED AND/OR FIELD OPS UTILIZED

All work will be performed in accordance with the provisions of this HASP, OSHA guidelines, and WESTON Standard Operating Procedures.

As warranted and noted in Section 2.1.5., Air monitoring in accordance with the Study Area-specific Community Air Monitoring Plan (CAMP), included as Attachment I.



TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.2 Task 2 Description

TASK 2: Groundwater monitoring well installation

EQUIPMENT REQUIRED/USED

Hollow-stem Auger Rig	pDR (dust monitoring)
Hard Hat	Water Level Meter
High-visibility vest (if required)	Mini-Rae (soil screening)
Safety boots	Nitrile gloves
Safety glasses	
Hearing protection	

POTENTIAL HAZARDS/RISKS

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
 What justifies risk level?
Encountering groundwater with the potential presence of SVOC and metal constituents at low concentrations.

Physical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
 What justifies risk level?
Work will primarily occur on private residential and public school property, and may extend into utility right-of-way areas.

Biological

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
 What justifies risk level?
Potential for ticks, bees, snakes, vegetation and small animals.

RADIOLOGICAL

☐ Hazard Present Risk Level: ☐ H ☐ M ☐ L
 What justifies risk level?
N/A

LEVELS OF PROTECTION/JUSTIFICATION

Level D - safety shoes, safety glasses, hard hat, high-visibility vest (if required).

SAFETY PROCEDURES REQUIRED AND/OR FIELD OPS UTILIZED

All work will be performed in accordance with the provisions of this HASP, OSHA guidelines, and WESTON Standard Operating Procedures.
As warranted and noted in Section 2.1.5., Air monitoring in accordance with the Study Area-specific Community Air Monitoring Plan (CAMP), included as Attachment I.



4.1 TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.3 Task 3 Description

TASK 3: Groundwater sampling

EQUIPMENT REQUIRED/USED

Nitrile gloves	Sample bottleware
Safety boots	Bailers
Safety glasses	String
High-visibility vest (if required)	Tubing
Hand tools	Mini-Rae (if required for headspace)
Water Level Meter	Generator
Electric pumps	Safety knife

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Sampling groundwater with the potential presence of SVOC and metal constituents at low concentrations.

Physical

☒ Hazard Present Risk Level: ☐ H ☒ M ☐ L

What justifies risk level?

Work will primarily occur on private residential and public school property, and may extend into utility right-of-way areas.

Biological

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Potential for ticks, bees, snakes, vegetation and small animals.

RADIOLOGICAL

☐ Hazard Present Risk Level: ☐ H ☐ M ☐ L

What justifies risk level?

N/A

LEVELS OF PROTECTION/JUSTIFICATION

Level D - safety shoes, safety glasses, hard hat, high-visibility vest (if required).

SAFETY PROCEDURES REQUIRED AND/OR FIELD OPS UTILIZED

All work will be performed in accordance with the provisions of this HASP, OSHA guidelines, and WESTON Standard Operating Procedures.

As warranted and noted in Section 2.1.5., Air monitoring in accordance with the Study Area-specific Community Air Monitoring Plan (CAMP), included as Attachment I.



4.1 TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.4 Task 4 Description

Task 4: Excavation and Test Pits, backfilling and sampling activities. Test pits will be approximately 1-1.5 feet in width; 4-7 feet in length, and to a maximum depth of 4 ft below ground surface (ft bgs). Observations will be made by field personnel from outside of the test pits. Trenches and test pits will not be entered at any point. All trenches and test pits will be backfilled immediately upon completion of logging/sampling activities. No areas of excavation will remain open overnight.

EQUIPMENT REQUIRED/USED

Construction Equipment (Mini-excavator / backhoe / front end loader)
Hard hat
Safety boots
Safety glasses
High-visibility vest (if required)

Hearing protection
Mini-Rae (soil screening)
Nitrile gloves

POTENTIAL HAZARDS/RISKS

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
What justifies risk level?
Soil sampling with the potential presence of metal constituents.

Physical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
What justifies risk level?
Activities include use of heavy machinery in residential and public areas.

Biological

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
What justifies risk level?
Potential for ticks, bees, snakes, vegetation and small animals.

RADIOLOGICAL

☐ Hazard Present Risk Level: ☐ H ☐ M ☐ L
What justifies risk level?
N/A

LEVELS OF PROTECTION/JUSTIFICATION

Level D - safety shoes, safety glasses, hard hat, high-visibility vest (if required).

SAFETY PROCEDURES REQUIRED AND/OR FIELD OPS UTILIZED

All work will be performed in accordance with the provisions of this HASP, OSHA guidelines, and WESTON Standard Operating Procedures.
As warranted and noted in Section 2.1.5., Air monitoring in accordance with the Study Area-specific Community Air Monitoring Plan (CAMP), included as Attachment I.



4.1 TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.5 Task 6 Description

Task 5 – Remediation (includes the removal of soils and layer of fill material constaining ash, brick and/or glass with depths ranging from 2 inches bgs to up to 4 ft bgs. May include removal and repair of impervious surfaces including walkways and driveways).

EQUIPMENT REQUIRED/USED

Jackhammer
Construction Equipment (Mini-excavator / backhoe / front end loader
Hard hat pDR (dust monitoring)
Safety boots Mini-Rae (soil screening)
Safety glasses Hearing protection
High-visibility vest (if required) Nitrile gloves

POTENTIAL HAZARDS/RISKS

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L

What justifies risk level?

Sampling soils with the potential presence of metal constituents.

Physical

☒ Hazard Present Risk Level: ☐ H ☐ M ☐ L

What justifies risk level?

Activities include use of heavy machinery in residential and public areas.

Biological

☒ Hazard Present Risk Level: ☐ H ☐ M ☐ L

What justifies risk level?

Potential for ticks, bees, snakes, vegetation and small animals.

RADIOLOGICAL

☐ Hazard Present Risk Level: ☐ H ☐ M ☐ L

What justifies risk level?

N/A

LEVELS OF PROTECTION/JUSTIFICATION

Level D - safety shoes, safety glasses, hard hat, high-visibility vest (if required).

SAFETY PROCEDURES REQUIRED AND/OR FIELD OPS UTILIZED

As warranted and noted in Section 2.1.5., Air monitoring in accordance with the Study Area-specific Community Air Monitoring Plan (CAMP), included as Attachment I.



4.2 PERSONNEL PROTECTION PLAN

Engineering Controls

Describe Engineering Controls used as part of Personnel Protection Plan:

Task(s)
4,5

Depth of excavations will be no more than 4 ft bgs

Administrative Controls

Describe Administrative Controls used as part of Personnel Protection Plan:

Task(s)

All Conduct daily safety briefings with contractors prior to performing any tasks to discuss safety hazards and controls taken to minimize or eliminate hazards.

All Study Area Air Monitoring plan (i.e., Community Air Monitoring Plan [CAMP]) (**ATTACHMENT I**).

Personal Protective Equipment

Action Levels for Changing Levels of Protection. Refer to Site Air Monitoring Program—Action Levels. Define Action Levels for up or down grade for each task:

Task(s)

All Tasks 1-5
Hard hat, safety glasses, safety shoes, hearing protection, high-visibility vest (as necessary), nitrile gloves (as necessary)

All PPE will be reviewed with each hazard analysis to ensure level of PPE is appropriate for scope of work

Description of Levels of Protection

Level D		Level D Modified	
Task(s): All		Task(s): NA	
☒ Head	Hard hat when near drilling rig, excavating equipment	☐ Head	
☒ Eye and Face	Safety Glasses	☐ Eye and Face	
☒ Hearing	Ear plugs in designated areas and during drilling activities	☐ Hearing	
☐ Arms and Legs Only		☐ Arms and Legs Only	
☒ Appropriate Work Uniform	Coveralls or long pants and appropriate shirt	☐ Whole Body	
☒ Hand – Gloves	Nitrile (as needed)	☐ Apron	
☒ Foot - Safety Boots	Steel-toed boots	☐ Hand - Gloves	
☐ Fall Protection		☐ Gloves	
☐ Flotation		☐ Gloves	
☐ Other		☐ Foot - Safety Boots	
		☐ Over Boots	



4.3 DESCRIPTION OF LEVELS OF PROTECTION	
Level C	Level B () or Level A ()
Task(s): NA ☐ Head ☐ Eye and Face ☐ Hearing ☐ Arms and Legs Only ☐ Whole Body ☐ Apron ☐ Hand – Gloves ☐ Gloves ☐ Gloves ☐ Foot - Safety Boots ☐ Outer Boots ☐ Boots (Other) ☐ Half Face ☐ Cart./Canister ☐ Full Face ☐ Cart./Canister ☐ PAPR ☐ Cart./Canister ☐ Type C ☐ Fall Protection ☐ Flotation ☐ Other	Task(s): NA ☐ Head ☐ Eye and Face ☐ Hearing ☐ Arms and Legs Only ☐ Whole Body ☐ Apron ☐ Hand - Gloves ☐ Gloves ☐ Gloves ☐ Foot - Safety Boots ☐ Outer Boots ☐ Boots (Other) ☐ SAR - Airline ☐ SCBA ☐ Comb. Airline/SCBA ☐ Cascade System ☐ Compressor ☐ Fall Protection ☐ Flotation ☐ Other



5. MONITORING PROGRAM



5.1 SITE OR PROJECT HAZARD MONITORING PROGRAM

5.1.1 Air Monitoring Instruments

Instrument Selection and Initial Check Record

Reporting Format: ☒ Field Notebook ☒ Field Data Sheets* ☐ Air Monitoring Log ☐ Trip Report ☐ Other

Instrument	Task No.(s)	Number Required	Number Received	Checked Upon Receipt	Comment	Initials
☐ RAD				☐		
☐ GM (Pancake)				☐		
☐ NaI (Micro R)				☐		
☐ ZnS (Alpha Scintillator)				☐		
☐ Other _____				☐		
☒ PID	1,2,4,5	1		☐		
☒ MiniRAE				☐		
☐ MultiRAE (LEL/O2/H2S/CO/PID)				☐		
☐ TVA 1000 (PID/FID)				☐		
☐ Other _____				☐		
☐ FID				☐		
☐ TVA 1000 (FID/PID)				☐		
☐ Other _____				☐		
☒ PDR 1000 (Particulate)	1,2,4,5	2		☐		
☐ Single Gas Meter (SGM)				☐		
Specify Chemical:				☐		
☐ Personal Sampling Pump				☐		
Specify Media:				☐		
☐ Bio-Aerosol Monitor				☐		
☐ Tubes/type: _____						
☐ Tubes/type: _____						
☐ Tubes/type: _____						
☐ Tubes/type: _____						



5.1 SITE OR PROJECT HAZARD MONITORING PROGRAM

5.1.1 Air Monitoring Instruments Calibration Record

[illegible]



5.2 SITE AIR MONITORING PROGRAM

Action Levels

These Action Levels, if not defined by regulation, are some percent (usually 50%) of the applicable PEL/TLV/REL. That number must also be adjusted to account for instrument response factors.

	Tasks	Action Level		Action
☐ Explosive or Flammable Atmosphere		Ambient Air Concentration	Confined Space Concentration	
		<10% LEL	0 to 1% LEL	Work may continue. Consider toxicity potential.
		10 to 25% LEL	1 to 10% LEL	Work may continue. Increase monitoring frequency.
		>25% LEL	>10% LEL	Work must stop. Ventilate area before returning.
☐ Oxygen		Ambient Air Concentration	Confined Space Concentration	
		<19.5% O ₂	<19.5% O ₂	Leave area. Re-enter only with self-contained breathing apparatus.
		19.5% to 25% O ₂	19.5% to 23.5% O ₂	Work may continue. Investigate changes from 21%.
		>25% O ₂	>23.5% O ₂	Work must stop. Ventilate area before returning.
☐ Radiation	3, Radiation screening related to XRF to be performed by selected subcontractor for XRF work	< 3 times background 3 times background to < 1 mR/hour		Continue work. Radiation above background levels (normally 0.01-0.02 mR/hr) signifies possible radiation source(s) present. Continue investigation with caution. Perform thorough monitoring. Consult with a Health Physicist.
		> 1 mrem/hour		Potential radiation hazard. Evacuate site. Continue investigation only upon the advice of Health Physicist.
☒ Organic Gases and Vapors	(1-5)*	2.5 units sustained above background per 15-minute period		Routine screening of subsurface soils only. (N/A)
☒ Inorganic Gases, Vapors, and Particulates	(1-5)*	100 µg/m ³ above background per 15-minute period		Continue work with dust suppression techniques. If levels exceed 150 µg/m ³ above background per 15-minute period. Stop work and re-evaluate dust suppression.

* Some activities will be performed in areas of known concentrations of constituents. Air monitoring for organic vapors may be suspended by the Field Safety Officer.



5.3 ACTION LEVELS

The organic gases and vapors action level of 2.5 parts per million (ppm) above background levels is used as an action level. No VOCs detected in soils or groundwater at concentrations greater than applicable standards in the Residential Area. However, the New York State Department of Environmental Conservation (NYSDEC) has used 5 ppm action level for which soil VOC sampling was required previously on this project (this activity is no longer required based on sample results indicating no detected VOCs greater than respective screening levels in the area). To be conservative an action level was set at ½ of this value.

The New York State Department of Environmental Conservation (NYSDEC) recommended action level for fugitive dust is 100 µg/m³ greater than background (measured at the upwind location) for a 15 minute time period).

Calculate Action Levels for constituents of potential concern using the most conservative OSHA Permissible Exposure Limits (PELs)
(www.osha.gov/dsg/annotated-pels/tablez-1.html)

Exposure limits are:

Arsenic: 0.01 mg/m³ (ACGIH 2016 TLV, 8-hr TWA)

Cadmium: 0.005 mg/m³ (Cal/OSHA PEL, 8-hr TWA)

Lead: 0.05 mg/m³ (ACGIH 2016 TLV, 8-hr TWA)

Action Level (AL) = [Exposure Limit (mg/m³) X 1,000,000] / [concentration of the contaminant (mg/kg)] X 2

Arsenic (average soil concentration = 13.5 mg/Kg)

AL = [0.001 X 1,000,000] / 13.5 X 2 = 10,000/27 = 370 µg/m³

Cadmium (average soil concentration = 6.4 mg/Kg)

AL = [0.005 X 1,000,000] / 6.4 X 2 = 5000/12.8 = 391 µg/m³

Lead (average soil concentration = 167.0 mg/Kg)

AL = [0.050 X 1,000,000] / 167.0 X 2 = 50,000/334 = 150 µg/m³

To be conservative, the Action Level for fugitive dust remains at the 100µg/m³ greater than background (measured at the upwind location) for a 15 min-time period (i.e., the Action Level set by the NYSDEC for the Community Air Monitoring Program included in Attachment I).



6. HOSPITAL INFORMATION



6.1 CONTINGENCIES

6.1.1 Emergency Contacts and Phone Numbers

Agency	Contact	Phone Number
WorkCare WESTON Medical Director WorkCare WESTON Program Administrator	Dr. Peter Greaney Heather Lind	From 6 am to 4:30 pm Pacific Time call 800-455-6155 and dial 0 for the Operator or ext. 475 for Heather Lind to request the on-call clinician.
After-Business Hours Contact (In Case of Emergency Only)		4:31 p.m. – 5:59 a.m. Pacific Time, all day Saturday, Sunday, and Holidays call 800-455-6155 Dial 3 to reach the after-hours answering service. Request that the service connect you with the on-call clinician or the on-call clinician will return your call within 30 minutes.
WESTON Corporate Environmental Health & Safety Director	Harold Hannah	(610) 701-3024; (267) 516-0274 (Cell)
WESTON Health & Safety Division Safety Manager	Thomas Drew	(610) 701-3677; (610) 368-0950 (Cell)
WESTON Health & Safety Local Safety Officer	Thomas Drew	(610) 701-3677; (610) 368-0950 (Cell)
Fire Department	911	911
Police Department	911	911
WESTON FSO Cell Phone	Rachel McLoughlin	(610) 701-3428; (484) 433-2377 (Cell)
WESTON PM Cell Phone	John Sontag	(610) 701-3679; (484) 678-6135 (Cell)
Client Site Phone	Weston Field Office	(607) 378-7991 (cell)
Site Telephone	Weston Field Office	(607) 378-7991 (cell)
Nearest Telephone	Weston Field Office	(607) 378-7991 (cell)
Poison Control		(800) 222-1222
Local Medical Emergency Facility(s) - LMF		
Name of Hospital: Guthrie Corning Hospital		
Address: 1 Guthrie Drive, Corning, NY 14830		Phone No.: 607-937-8674
Name of Contact:		Phone No.:
Type of Service: ☒ Physical trauma only ☐ Chemical exposure only ☐ Physical trauma and chemical exposure ☐ Available 24 hours	Route to Hospital: (See Attached)	Travel time from site: 9 Minutes Distance to hospital: 5.2 Miles Name/no. of 24-hr ambulance service: 911

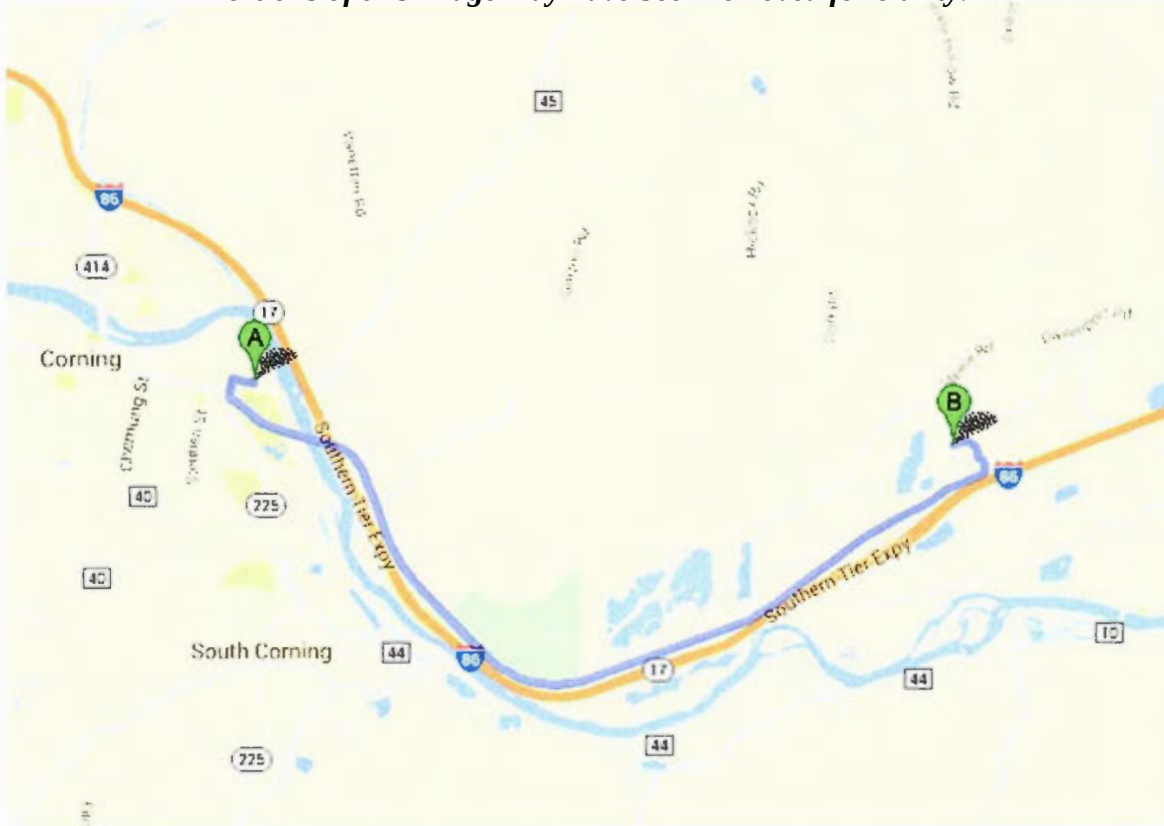


Secondary or Specialty Service Provider		
Name of Hospital:		
Address:		Phone No.:
Name of Contact:		Phone No.:
Type of Service: ☐ Physical trauma only ☐ Chemical exposure only ☐ Physical trauma and chemical exposure ☐ Available 24 hours	Route to Hospital (see attached):	Travel time from site: Distance to hospital: Name/no. of 24-hr ambulance service: /

See reporting an incident in Attachment F.

6.1.2 Hospital Map

This map is subject to Google's Terms of Service, and Google is the owner of rights therein. Portions of this image may have been removed for clarity.



A 59 Canisteo St, Corning, NY 14830

1. Head **south** on **Canisteo St** toward **Woodview Ave**

go 82 ft
total 82 ft
2. Take the 1st right onto **Woodview Ave**

go 0.1 mi
total 0.1 mi
3. Take the 1st left onto **Conhocton St**

go 495 ft
total 0.2 mi
4. Take the 2nd left onto **NY-352 E/E Corning Rd/Denison Pkwy E**
Continue to follow NY-352 E/E Corning Rd
About 5 mins

go 3.8 mi
total 4.0 mi
5. Continue straight onto **E Corning Rd**
Destination will be on the left
About 2 mins

go 1.2 mi
total 5.2 mi

B **Guthrie Corning Hospital**
1 Guthrie Drive, Corning, NY 14830



6.1 CONTINGENCIES				
6.1.3 Response Plans				
Medical - General Provide first aid, if trained; assess and determine need for further medical assistance. Transport or arrange for transport after appropriate decontamination. LMF = Local Medical Facility	First Aid Kit: ☒ Yes ☐ No Blood Borne Pathogens Kit: ☒ Yes ☐ No	Type Appropriate sized ANSI-approved Type III Kit, plus BBP	Location In Vehicle near work area	Special First-Aid Procedures: Cyanides on-site ☐ Yes ☒ No If yes, contact LMF. Do they have antidote kit? ☐ Yes ☐ No
	Eyewash required ☒ Yes ☐ No	Type Saline Solution	Location Field vehicles	HF on-site ☐ Yes ☒ No If yes, need neutralizing ointment for first-aid kit. Contact LMF.
	Shower required ☐ Yes ☒ No	Type	Location	
Plan for Response to Spill/Release		Plan for Response to Fire/Explosion		Fire Extinguishers
In the event of a spill or release, ensure safety, assess situation, and perform containment and control measures, as appropriate. ½œ@re safety, assess situation, and perform containment and control measures as appropriate:	a. Cleanup per SDSs if small; or sound alarm, call for assistance, notify Emergency Coordinator (R. McLoughlin) b. Evacuate to pre-determined safe place c. Account for personnel d. Determine if team can respond safely e. Mobilize per Site Spill Response Plan	In the event of a fire or explosion, ensure personal safety, assess situation, and perform containment and control measures, as appropriate:	a. Sound alarm and call for assistance, notify Emergency Coordinator b. Evacuate to predetermined safe place c. Account for personnel d. Use fire extinguisher <u>only if safe and trained</u> in its use e. Stand by to inform emergency responders of materials and conditions	Type/Location <u>ABC/Vehicle</u> / / / / / /
Description of Spill Response Gear	Location	Description (Other Fire Response Equipment)		Location



7. DECONTAMINATION PLAN



7.1 GENERAL DECONTAMINATION PLAN

Personnel Decontamination

Consistent with the levels of protection required, step-by-step procedures for personnel decontamination for each level of protection are attached.

Level D PPE with used PPE properly disposed on-site

Levels of Protection Required for Decontamination Personnel

The levels of protection required for personnel assisting with decontamination will be:

☐

Level B

☐

Level C

☒

Level D

Modifications include:

Disposition of Decontamination Wastes

Drill cuttings and other waste soil/water generated during characterization activities will be containerized daily in 55-gallon drums or other appropriate containers (as described in the Study Area Work Plan). The filled containers will be staged in a secure, designated area (within the fenced area). The waste soil and waste water will be properly disposed by a certified waste handler, in accordance with analytical results.

All IDWs will be contained in 55-gallon drums and staged inside the fenced area. In addition, all drilling and excavating equipment will be transported to the fenced area for overnight storage.

Liquid drums will be placed inside a secondary containment structure, and staged within the fenced area. Liquid drums will be emptied of their contents as needed by a certified waste handler.

Equipment Decontamination

A procedure for decontamination steps required for non-sampling equipment and heavy machinery follows:

Heavy equipment will be decontaminated in accordance with the decontamination SOP included in the Study Area Work Plan.

Temporary decontamination structures will be staged inside the fenced area. The structures will be equipped with side walls, in order to contain all fluids. All decontamination fluids will be pumped daily into 55-gallon drums, no more than $\frac{3}{4}$ full, and staged within secondary containment. The total volume of fluid IDWs is expected to be low. Liquid drums will be emptied of their contents as needed by a certified waste handler.

Decontamination procedures are detailed in SOP D.1 (included as Attachment J).

Sampling Equipment Decontamination

Sampling equipment will be decontaminated in accordance with the following procedure:

Sampling equipment is anticipated to be single-use only materials (includes glass jars, nitrile gloves, plastic paint trays, disposable spoons and ziplock bags). No decontamination activities will be required for these items.

Reusable sampling equipment will be decontaminated in accordance with SOP D.1, which is included as Attachment J.



7.2 LEVEL D DECONTAMINATION PLAN	
Check indicated functions or add steps, as necessary:	
Function	Description of Process, Solution, and Container
☐ Segregated equipment drop	
☐ Boot cover and glove wash	
☐ Boot cover and glove rinse	
☐ Tape removal - outer glove and boot	
☐ Boot cover removal	
☒ Outer glove removal	Disposable PPE (nitrile gloves) will be discarded in designated areas
HOTLINE	
☐ Suit/safety boot wash	
☐ Suit/boot/glove rinse	
☐ Safety boot removal	
☐ Suit removal	
☐ Inner glove wash	
☐ Inner glove rinse	
☐ Inner glove removal	
☐ Inner clothing removal	
CONTAMINATION REDUCTION ZONE (CRZ)/SAFE ZONE BOUNDARY	
☐ Field wash	
☐ Redress	
Disposal Plan, End of Day: Single-use PPE (nitrile gloves, ear-plugs) will be disposed of in approved containers at the completion of a work task. Non-disposable PPE will be wiped clean (hard hats, safety boots, safety glasses) if visibly soiled. In general, activities in Level D will not require personnel decontamination	
Disposal Plan, End of Week: NA	
Disposal Plan, End of Project: Disposable sampling equipment will be disposed in accordance with applicable waste management procedure. Sampling equipment that is not dedicated disposable equipment will be decontaminated in accordance with procedures in the SOPs.	



7.3 LEVEL C DECONTAMINATION PLAN

Check indicated functions or add steps, as necessary:

Function	Description of Process, Solution, and Container
☐ Segregated equipment drop	
☐ Boot cover and glove wash	
☐ Boot cover and glove rinse	
☐ Tape removal - outer glove and boot	
☐ Boot cover removal	
☐ Outer glove removal	
HOTLINE	
☐ Suit/safety boot wash	
☐ Suit/boot/glove rinse	
☐ Safety boot removal	
☐ Suit removal	
☐ Inner glove wash	
☐ Inner glove rinse	
☐ Face piece removal	
☐ Inner glove removal	
☐ Inner clothing removal	
CONTAMINATION REDUCTION ZONE (CRZ)/SAFE ZONE BOUNDARY	
☐ Field wash	
☐ Redress	
Disposal Plan, End of Day:	
N/A	
Disposal Plan, End of Week:	
Disposal Plan, End of Project:	



7.4 LEVEL B () or Level A () DECONTAMINATION PLAN	
Check indicated functions or add steps, as necessary:	
Function	Description of Process, Solution, and Container
☐ Segregated equipment drop	
☐ Boot cover and glove wash	
☐ Boot cover and glove rinse	
☐ Tape removal - outer glove and boot	
☐ Boot cover removal	
☐ Outer glove removal	
HOTLINE	
☐ Suit/safety boot wash	
☐ Suit/SCBA/boot/glove rinse	
☐ Safety boot removal	
☐ Remove SCBA backpack without disconnecting	
☐ Splash suit removal	
☐ Inner glove wash	
☐ Inner glove rinse	
☐ SCBA disconnect and face piece removal	
☐ Inner glove removal	
☐ Inner clothing removal	
CONTAMINATION REDUCTION ZONE (CRZ)/SAFE ZONE BOUNDARY	
☐ Field wash	
☐ Redress	
Disposal Plan, End of Day: N/A	
Disposal Plan, End of Week: 	
Disposal Plan, End of Project: 	



8. TRAINING AND BRIEFING TOPICS/SIGN OFF SHEET



8.1 TRAINING AND BRIEFING TOPICS

The following items will be covered at the site-specific training meeting, daily or periodically.

☒ Site characterization and analysis, Sec. 3.0, 29 CFR 1910.120 I	☐ Level A
☒ Physical hazards	☐ Level B
☒ Chemical hazards	☐ Level C
☒ Animal bites, stings, and poisonous plants	☒ Level D
☐ Etiologic (infectious) agents	☒ Monitoring, 29 CFR 1910.120 (h)
☒ Site control, 29 CFR 1910.120 d	☒ Decontamination, 29 CFR 1910.120 (k)
☒ Engineering controls and work practices, 29 CFR 1910.120 (g)	☒ Emergency response, 29 CFR 1910.120 (l)
☒ Heavy machinery	☒ Elements of an emergency response, 29 CFR 1910.120 (l)
☐ Forklift	☒ Procedures for handling site emergency incidents, 29 CFR 1910.120 (l)
☒ Backhoe	☒ Off-site emergency response, 29 CFR 1910.120 (l)
☒ Equipment	☒ Handling drums and containers, 29 CFR 1910.120 (j)
☒ Tools	☐ Opening drums and containers
☐ Ladder, 29 CFR 1910.25.26.26 + 29 CFR 1926.1053	☐ Electrical material handling equipment
☒ Overhead and underground utilities	☐ Radioactive waste
☐ Scaffolds	☐ Shock-sensitive waste
☐ Structural integrity	☐ Laboratory waste packs
☐ Unguarded openings - wall, floor, ceilings	☐ Sampling drums and containers
☐ Pressurized air cylinders	☒ Shipping and transport, 49 CFR 172.101, IATA
☒ Personal protective equipment, 29 CFR 1910.120 (g); 29 CFR 1910.134	☐ Tank and vault procedures
☐ Respiratory protection, 29 CFR 1910.120 (g); ANSI Z88.2	☐ Illumination, 29 CFR 1926.26
☐ Working over water FLD-19	☐ Sanitation, 29 CFR 1926.27
☐ Boating safety FLD-18	☒ Proper lifting techniques
☒ Heat Stress / Cold Stress	☒ Lead, Arsenic, Cadmium exposure training



8.2 HEALTH AND SAFETY PLAN APPROVAL/SIGNOFF FORM

Site Name: Study Area, Corning, New York

WO#: 02005.056.002.0001

Address: Located in Corning, New York on the north bank of the Chemung River (see Figure 1).

I understand, agree to, and will conform with the information set forth in this Health and Safety Plan (and attachments) and discussed in the personnel health and safety briefing(s).

Name

Signature

Date _____

[illegible]



ATTACHMENT A

CHEMICAL CONTAMINANTS DATA SHEETS



ATTACHMENT B
SAFETY DATA SHEETS
(ATTACH SDS)



ATTACHMENT C

SAFETY PROCEDURES/FIELD OPERATING PROCEDURES (FLD OPS)

In lieu of attaching individual copies of FLDs, the site safety officer or his designee may elect to maintain an electronic copy of the WESTON Corporate Environmental Compliance, Health, and Safety Program Manual (including all FLDs) on site in an electronic format. The most recent version of the CEHS Program Manual and supporting documents are located at:

<http://portal/services/EHS/SitePages/CEHSProgramElements.aspx>



ATTACHMENT D

HAZARD COMMUNICATION PROGRAM



SITE-SPECIFIC HAZARD COMMUNICATION PROGRAM

Location-Specific Hazard Communication Program/Checklist

To ensure an understanding of and compliance with the Hazard Communication Standard, WESTON will use this checklist/document (or similar document) in conjunction with the WESTON Written Hazard Communication Program as a means of meeting site- or location-specific requirements.

While responsibility for activities within this document reference the WESTON Safety Officer (SO), it is the responsibility of all personnel to ensure compliance. Responsibilities under various conditions can be found within the WESTON Written Hazard Communication Program.

To ensure that information about the dangers of all hazardous chemicals used by WESTON is known by all affected employees, the following Hazard Communication Program has been established. All affected personnel will participate in the Hazard Communication Program. This written program, as well as WESTON's Corporate Hazard Communication Program, will be available for review by any employee, employee representative, representative of OSHA, NIOSH, or any affected employer/employee on a multi-employer site.

- ☐ Site or other location name/address: Study Area, Corning, NY
- ☐ Site/Project/Location Manager: John Sontag
- ☐ Site/Location Safety Officer: Rachel McLoughlin
- ☐ List of chemicals compiled, format: ☒ HASP ☐ Other: _____
- ☐ Location of SDS files: Attached
- ☐ Training conducted by: Name: TBD Date: _____
- ☐ Indicate format of training documentation: ☒ Field Log ☐ Other: _____
- ☐ Client briefing conducted regarding hazard communication: _____
- ☐ If multi-employer site (client, subcontractor, agency, etc.), indicate name of affected companies: _____
- ☐ Other employer(s) notified of chemicals, labeling, and SDS information: _____
- ☐ Has WESTON been notified of other employer's or client's hazard communication program(s), as necessary? ☐ Yes ☒ No

List of Hazardous Chemicals

A list of known hazardous chemicals used by WESTON personnel must be prepared and attached to this document or placed in a centrally identified location with the SDSs. Further information on each chemical may be obtained by reviewing the appropriate SDS. The list will be arranged to enable cross-reference with the SDS file and the label on the container. The SO or Location Manager is responsible for ensuring the chemical listing remains up-to-date.

Container Labeling

The WESTON SO will verify that all containers received from the chemical manufacturer, importer, or distributor for use on-site are clearly labeled.

The SO is responsible for ensuring that labels are placed where required and for comparing SDSs and other information with label information to ensure correctness.



Safety Data Sheets (SDSs)

The SO is responsible for establishing and monitoring WESTON's SDS program for the location. The SO will ensure that procedures are developed to obtain the necessary SDSs and will review incoming SDSs for new or significant health and safety information. He/she will see that any new information is passed on to the affected employees. If an SDS is not received at the time of initial shipment, the SO will call the manufacturer and have an SDS delivered for that product in accordance with the requirements of WESTON's Written Hazard Communication Program.

A log for, and copies of, SDSs for all hazardous chemicals in use will be kept in the SDS folder at a location known to all site workers. SDSs will be readily available to all employees during each work shift. If an MSDS is not available, immediately contact the WESTON SO or the designated alternate. When a revised SDS is received, the SO will immediately replace the old SDS.

Employee Training and Information

The SO is responsible for the WESTON site-specific personnel training program. The SO will ensure that all program elements specified below are supplied to all affected employees.

At the time of initial assignment for employees to the work site, or whenever a new hazard is introduced into the work area, employees will attend a health and safety meeting or briefing that includes the information indicated below.

- Hazardous chemicals present at the work site.
- Physical and health risks of the hazardous chemicals.
- The signs and symptoms of overexposure.
- Procedures to follow if employees are overexposed to hazardous chemicals.
- Location of the SDS file and Written Hazard Communication Program.
- How to determine the presence or release of hazardous chemicals in the employee's work area.
- How to read labels and review SDSs to obtain hazard information.
- Steps WESTON has taken to reduce or prevent exposure to hazardous chemicals.
- How to reduce or prevent exposure to hazardous chemicals through the use of controls procedures, work practices, and personal protective equipment.
- Hazardous, non-routine tasks to be performed (if any).
- Chemicals within unlabeled piping (if any).

Hazardous Non-routine Tasks

When employees are required to perform hazardous non-routine tasks, the affected employee(s) will be given information by the SO about the hazardous chemicals he or she may use during such activity. This information will include specific chemical hazards, protective and safety measures the employee can use, and steps WESTON is using to reduce the hazards. These steps include, but are not limited to, ventilation, respirators, presence of another employee, and emergency procedures.

Multi-Employer Work Sites

It is the responsibility of the SO to provide other employers with information about hazardous chemicals imported by WESTON to which their employees may be exposed, along with suggested safety precautions. It is also the responsibility of the SO and the Site Manager to obtain information about hazardous chemicals used by other employers to which WESTON employees may be exposed. WESTON's chemical listing will be made available to other employers, as requested. SDSs will be available for viewing, as necessary.

The location, format, and/or procedures for accessing SDS information must be relayed to affected employees.



ATTACHMENT E

AIR SAMPLING DATA SHEETS



AIR MONITORING PROGRAM								
Field Data Sheets								
Location:								
% LEL	% O ₂	PID (units)	FID (units)	Aerosol Monitor (mg/m ³)	GM: Shield Probe/ Thin Window		NaI (uR/hr)	ZnS (cpm)
					mR/hr	cpm		
Monitox (ppm)				Detector Tube(s)				
Sound Levels (dBA)		Illumination	pH	Other	Other	Other	Other	Other
Location:								
% LEL	% O ₂	PID (units)	FID (units)	Aerosol Monitor (mg/m ³)	GM: Shield Probe/ Thin Window		NaI (uR/hr)	ZnS (cpm)
					mR/hr	cpm		
Monitox (ppm)				Detector Tube(s)				
Sound Levels (dBA)		Illumination	pH	Other	Other	Other	Other	Other

[illegible]

QA by:

Date:



ATTACHMENT F INCIDENT REPORTING



Windows Internet Explorer window titled "Welcome to NOITrack.: - Windows Internet Explorer". The address bar shows "http://prdnnet/noitrack/IncidentInfo.aspx". The browser interface includes a menu bar (File, Edit, View, Favorites, Tools, Help), a search bar, and a toolbar with buttons for Google, Search, Bookmarks, Check, AutoFill, and Sign In. The NOITrack application is displayed within the browser. The header features the "NOITrack" logo and a navigation bar with links: Open NOI's, Search, Add New Incident, Reports, Admin, Help, and Blog. The main content area has tabs for Incident Info, Individual Data, Investigation, and File Attachment. The "Incident Info" tab is active, showing a form with a "Near Incident" checkbox and a note: "Fields marked with * are required". The form is divided into four columns: Security, Safety, Computer, and Other. The Security column includes checkboxes for Threat or Intimidation, Act of Violence, Theft, Vandalism, Violation of Company or Government Security Requirements, and Other Security. The Safety column includes checkboxes for Vehicle, Injury, Illness, Exposure, and Other Safety. The Computer column includes checkboxes for Computer/Technology and Other. The Other column includes checkboxes for Environmental, Property/Equipment Damage, Regulatory Agency, and Other. Below the columns is a text area for a description, with a note: "Note: This description is limited to 255 characters. If more information is required, add the information in the submitted description." At the bottom, there are fields for Date of Incident (with a calendar icon) and Time of Incident (with AM/PM dropdowns), and checkboxes for Unknown Date and Unknown Time. The status bar at the bottom shows "Done", "Local intranet", and "100%".

Please go to NOITrack using the following link to complete incident reporting. If you are in the field and do not have access to NOITrack, please contact someone in your office to do the reporting for you.

<http://asweb/noitrack/IncidentInfo.aspx>

Questions can be directed to Susan Hipp-Ludwick at 610.701.3046.



ATTACHMENT G

TRAFFIC CONTROL PLAN



ATTACHMENT G
Traffic Control Plan

A formal Traffic Control Plan is not required for the Pre-Design Investigation work to be conducted in the Study Area. If needed, WESTON will obtain the appropriate traffic control permits from the New York State Department of Transportation.

All WESTON personnel working in or proximal to roadways will be equipped with Level D PPE, including high-visibility vests, hard hats, safety boots and safety glasses. A track-mounted Geoprobe rig will be used to advance soil borings on both public and private properties. No obstructions to roadways are anticipated for the Pre-Design Investigation phase; however, obstructions to sidewalks may occur during drilling activities. All obstructions will be marked with traffic cones and/or flagging to prevent pedestrians from entering designated work areas. In addition, traffic cones will be placed around the rig and/or trailer if equipment is parked on the side of any roadways.

A spotter will be used for roadway and/or pedestrian traffic whenever the rig is relocated throughout the Study Area. As much as possible, heavy equipment will be directed along the sides of the roadways to avoid obstructions in right-of-way mains. The drilling contractor must use extreme caution when relocating the rig to avoid property damage and ensure the safety of pedestrians.



ATTACHMENT H

ENVIRONMENTAL HEALTH & SAFETY INSPECTION CHECKLIST



ENVIRONMENTAL HEALTH AND SAFETY INSPECTION CHECKLIST

Project Name: _____

Inspector: _____

Submit to: _____

Date: _____



THE WESTON SITE APPEARANCE

YES	NO		COMMENT
☐	☐	Is the site secured to prevent inadvertent, unnecessary, or unauthorized access? Are gates closed and locked at any time that the access point is not occupied or visible to site workers?	
☐	☐	Are access points posted with signs to indicate client and end-user client name, WESTON's name and logo, names of other contractors and sub-contractors, project name and location, and appropriate safety messages?	
☐	☐	Are required postings in place (e.g., Labor Poster, Emergency Phone Numbers, Site Map, etc.)?	
☐	☐	Are site trailers tied down per local code and provided with stairs that have a landing platform with guard and stair railings?	
☐	☐	Is a Site Safety file system established in the office to maintain records required by applicable safety regulations	
☐	☐	Is the Health and Safety Plan (HASP) or Accident Prevention Plan (APP) amended as scope of work changes, hazards are discovered or eliminated or if risk change?	
☐	☐	Is the Site Safety Plan and the Safety Officers Field Manual on site?	
☐	☐	Is new employee indoctrination provided?	
☐	☐	Have site Rules been provided, discussed and signed off on by all employees	
☐	☐	Incident Reporting procedure explained to all?	
☐	☐	Is site management trained in the WESTON (and client as applicable) Incident Reporting system?	
☐	☐	Are NOI and Supplemental Report forms and OSHA 300 Log available on site?	
☐	☐	Is Site Management aware of the Case Management and Incident Investigation Procedures?	
☐	☐	Is there a list of preferred provider medical facilities available?	
☐	☐	Has the "Inspection By A Regulatory Agency" procedure been reviewed by all site management?	
☐	☐	Will Competent Persons be required because of activities to be performed, equipment to be used or hazards to be encountered?	

POLICIES

YES	NO		COMMENT
☐	☐	Each individual employee is aware that he or she responsible for complying with applicable safety requirements, wearing prescribed safety equipment and preventing avoidable accidents.	
☐	☐	Do employees understand that they will wear clothing suitable for existing weather and work conditions and the minimum work uniform will include long pants, sleeved work shirts, protective footwear, hard hat, and safety glasses unless otherwise specified via the HASP.	
☐	☐	Are employees provided safety and health training to enable them to perform their work safely? Is all training documented to indicate the date of the session, topics covered, and names of participants?	
☐	☐	Safety meetings are conducted daily. The purpose of the meetings are to review past activities, review pertinent tailgate safety topics and establish safe working procedures for anticipated hazards encountered during the day.	
☐	☐	Training has been provided to all personnel regarding handling of emergency situations that may arise from the activity or use of equipment on the project.	
☐	☐	Employees/contractors are informed and understand that they may not be under the influence of alcohol, narcotics, intoxicants, or similar mind-altering substances at any time. Employees found under the influence of or consuming such substances will be immediately removed from the job site.	
☐	☐	Site workers and operators of any equipment or vehicles are able to read and understand the signs, signals, and operating instructions of their use.	
☐	☐	Have contractors performing work provided copies of relevant documentation (such as medical fit-for-duty, training certificates, fit-tests, etc.) prior to initiation of the project?	



SANITATION
29 CFR 1926 Subparts C, D. EM 385-1-1, Section 2

YES	NO		COMMENT
☐	☐	Is an adequate supply of drinking water provided? Is potable/drinking water labeled as such? Are there sufficient drinking cups provided?	
☐	☐	Are there a sufficient number of toilets?	
☐	☐	Are washing facilities readily available and appropriate for the cleaning needs?	
☐	☐	Are washing facilities kept sanitary with adequate cleansing and drying materials?	
☐	☐	Waste is secured so as not to attract rodents, insects, or other vermin?	
☐	☐	Is an effective housekeeping program established and implemented?	

ACCIDENT PREVENTION SIGNS, TAGS, LABELS, SIGNALS, AND PIPING SYSTEM IDENTIFICATION
29 CFR 1926 Subpart G. EM 385-1-1, Section 8

YES	NO		COMMENT
☐	☐	Are signs, tags, and labels provided to give adequate warning and caution of hazards and instruction/directions to workers and the public?	
☐	☐	Are all employees informed as to the meaning of the various signs, tags, and labels used in the workplace and what special precautions are required?	
☐	☐	Are construction areas posted with legible traffic signs at points of hazard?	
☐	☐	Are signs required to be seen at night lighted or reflectorized?	
☐	☐	Tags contain a signal word ("danger" or "caution") and a major message to indicate the specific hazardous condition or the instruction to be communicated to the employee. Tags follow requirements as outlined in 29 CFR 1926.200.	

MEDICAL SERVICES AND FIRST AID
29 CFR 1926 Subparts C, D. EM 385-1-1, Section 3

YES	NO		COMMENT
☐	☐	Is a local medical emergency facility (LMEF) identified in the HASP or APP?	
☐	☐	Has the LMEF been visited to verify the directions and establish contacts?	
☐	☐	Has site management reviewed WESTON's incident management procedures?	
☐	☐	Have clinics and specialists that will help WESTON manage injuries and illnesses been identified?	
☐	☐	Is there at least two (2) people certified in First Aid and CPR?	
☐	☐	Are first aid kits available at the command post and appropriate remote locations?	
☐	☐	Are first Aid Kits and Eyewash/Safety Showers inspected weekly?	
☐	☐	Are 15 minute eyewash/safety showers in place if required?	



FIRE PREVENTION AND PROTECTION
29 CFR 1926 Subpart F. EM 385-1-1, Section 9

YES	NO		COMMENT
☐	☐	Is an Emergency Response and Contingency Plan in place?	
☐	☐	Are emergency phone numbers posted?	
☐	☐	Are fire extinguishers selected and provided based on the types of materials and potential fire classes in each area?	
☐	☐	Are fire extinguishers provided in each administrative and storage trailer, within 50 ft but no closer than 25 ft of any fuel or flammable liquids storage, on welding and cutting equipment, on mechanical equipment?	
☐	☐	Are fire extinguishers checked daily and inspected monthly?	
☐	☐	Do site personnel know the location of fire extinguishers and how to use them?	
☐	☐	Are flammable and combustible liquids stored in approved containers?	
☐	☐	Safety cans are used for dispensing flammable or combustible liquids in 5 gallon or less volumes.	
☐	☐	Are flammable and combustible liquids stored in flammable storage cabinets or appropriate storage areas?	
☐	☐	Are flammable materials separated from oxidizers by at least 20 feet (or 5 foot tall, ½ -hour rated fire wall) when in storage?	
☐	☐	Are fuel storage tanks double walled or placed in a lined berm?	
☐	☐	Spills are cleaned up immediately and wastes are disposed of properly.	
☐	☐	Combustible scrap, debris, and waste material (oily rags) are stored in closed metal containers and disposed of promptly.	
☐	☐	Vehicle fueling tanks are grounded and bonding between the tank and vehicle being fueled is provided?	
☐	☐	LPG is stored, handled, and used according to OSHA regulations 29 CFR 1926.	
☐	☐	LPG cylinders are not stored indoors.	
☐	☐	Is a hot work permit program in place? See WESTON FLD-36	
☐	☐	Is smoking limited to specific areas, prohibited in flammable storage areas and are signs posted to this effect?	



HAZARDOUS SUBSTANCES, AGENTS, AND ENVIRONMENTS
29 CFR 1926 Subparts D, Z. EM 385-1-1, Sections 6, 28

YES	NO		COMMENT
☐	☐	Are operations, materials and equipment evaluated to determine the presence of hazardous contaminants or if hazardous agents could be released in the work environment?	
☐	☐	Are SDS for substances made available at the work-site when any hazardous substance is procured, used, or stored?	
☐	☐	Are all containers and piping containing hazardous substances labeled appropriately?	
☐	☐	Is there an inventory of hazardous substances?	
☐	☐	Is there a site Specific Hazard Communication Program?	
☐	☐	Spill kits appropriate for the hazardous materials present are on site and their location is known to spill responders.	
☐	☐	Is disposal of excess hazardous chemicals performed according to WESTON's guidelines and RCRA regulations?	
☐	☐	Before initiation of activities where there is an identified asbestos or lead hazard, is there a written plan detailing compliance with OSHA and EPA asbestos or lead abatement requirements? Does the plan comply with state and local authority, and USACE requirements, as applicable?	
☐	☐	Are personnel trained and provided with protection against hazards from animals, poisonous plants, and insects?	

CONFINED SPACES
29 CFR 1910 Subpart J. EM 385-1-1, Section 6

YES	NO		COMMENT
☐	☐	Is there a Confined Space Entry Program in place?	
☐	☐	Are the confined Spaces identified and labeled?	
☐	☐	Will the Confined Spaces be entered?	
☐	☐	Is appropriate entry documentation used and on-file?	



PERSONAL PROTECTIVE AND SAFETY EQUIPMENT, RESPIRATORY AND FALL PROTECTION
29 CFR 1926 Subparts D, E, M. EM 385-1-1, Section 5

YES	NO		COMMENT
☐	☐	Do employees understand that the minimum PPE is hard hat, safety glasses with side shields and safety shoes or boots and that long pants and a sleeved shirt are required?	
☐	☐	Has the SSHC reviewed the PPE requirements in the HASP against actual site conditions and certified that the PPE is appropriate? (see Field Manual, PPE Program)	
☐	☐	PPE is inspected, tested and maintained in serviceable and sanitary condition as recommended by the manufacturer. Is defective or damaged equipment taken out of service and repaired or replaced?	
☐	☐	Are workers trained in the use of the PPE required?	
☐	☐	Are personnel exposed to vehicular or equipment traffic, including signal persons, spotters or inspectors required to vests or apparel marked with a reflective or high visibility material?	
☐	☐	Is there a noise hazard? If yes, hearing protection will be required.	
☐	☐	Is there a splash or splatter hazard? Face shields or goggles will be required.	
☐	☐	Will personnel be working in or over water? Personnel Floatation devices will be required.	
☐	☐	Is there a welding hazard? Welding helmet and leathers will be required. Is there a cutting torch hazard? Goggles and protective clothing will be required.	
☐	☐	Is each person on a walking/working surface with an unprotected side or edge which is 6 feet (1.8 m) or more above a lower level protected from falling by the use of guardrail systems, safety net systems or personal fall arrest systems? See WESTON FLD 25 (Note General Industry standard is four feet).	
☐	☐	Guardrail systems are used as primary protection whenever feasible. Guardrail construction meets criteria in 29 CFR 1926.502(b).	
☐	☐	Personal fall arrest systems (PFAS) are inspected and appropriate for use.	
☐	☐	Ropes and straps (webbing) used in lanyards, lifelines, and strength components of body belts and body harnesses are from synthetic fibers.	
☐	☐	Safety nets and safety net installations are constructed, tested and used according to 29 CFR 1926.502.c	
☐	☐	Is respirator use required? See WESTON Respiratory Protection Program	
☐	☐	Persons using respiratory protection have been successfully medically cleared, trained, and fit tested.	
☐	☐	Respirators are used according to the manufacturer's instructions, regulatory requirements, selection criteria, and health and safety plan provisions.	
☐	☐	For Level C operations with organic vapor contamination, is the cartridge change-out schedule documented?	
☐	☐	Is breathing certified as Grade D, or better, and certification available on-site?	



MACHINERY AND MECHANIZED EQUIPMENT
29 CFR 1926 Subparts N, O, CC and DD. EM 385-1-1, Sections 16, 17, 18

YES	NO		COMMENT
☐	☐	Are inspections of machinery by a competent person established?	
☐	☐	Is equipment inspected daily before its next use?	
☐	☐	Equipment inspection reports are reviewed, followed-up on negative findings and records of inspections are maintained?	
☐	☐	Machinery or equipment found to be unsafe is taken out of service until the unsafe condition has been corrected.	
☐	☐	Is there a preventive maintenance program established?	
☐	☐	Are operators of equipment qualified and authorized to operate?	
☐	☐	Is all self-propelled construction and industrial equipment equipped with a reverse signal alarm?	
☐	☐	Are seats or equal protection provided for each person required to ride on equipment. Are seatbelts installed and worn on motor vehicles, as appropriate.	
☐	☐	All equipment with windshields is equipped with powered wipers. If fogging or frosting is possible, operable defogging or defrosting devices are required.	
☐	☐	Internal combustion engines are not operated in enclosed areas unless adequate ventilation is made. Air monitoring is conducted to assure safe working conditions.	
☐	☐	Is each bulldozer, scraper, dragline, crane, motor grader, front-end loader, mechanical shovel, backhoe, or similar equipment equipped with at least one dry chemical or carbon dioxide fire extinguisher with a minimum rating of 5-B:C?	
☐	☐	Will cranes or other lifting devices be used? If so, are the following documents available on site: 1) a copy of the operating manual, 2) load rating chart, 3) log book, 4) a copy of the last annual inspection and 5) the initial on-site inspection?	
☐	☐	Do operators have certificates of training to operate the type of crane(s) to be used?	
☐	☐	Is a signal person provided when the point of operation is not in full view of the vehicle, machine, or equipment operator? When manual (hand) signals are used, is only one person designated to give signals to the operator?	
☐	☐	Signal persons back one vehicle at a time. While under the control of a signal person, drivers do not back or maneuver until directed. Drivers stop if contact with the signal person is lost.	
☐	☐	Is a critical lift plan prepared by a competent person whenever: a lift is not routine, or a lift exceeds 75% of a crane's capacity, a lift results in the load being out of the operator's line of sight, or a lift involves more than one crane, a man basket is used, or the operator believes there is a need for a critical lift plan.	
☐	☐	Fork Lifts (Powered Industrial Trucks) - Will forklifts be used on site?	
☐	☐	All forklifts meet the requirements of design, construction, stability, inspection, testing, maintenance, and operation as indicated in ANSI/ASME B56.1 Safety Standards for Low Lift and High Lift Trucks.	
☐	☐	Do forklift operators have certificates of training?	
☐	☐	Are pile driving operations conducted according to EM 385-1-1, Section 16.L?	
☐	☐	Is drilling equipment operated, inspected, and maintained as specified in the manufacturer's operating manual? Is a copy of the manual available at the work-site? See also the Drilling Safety Guide in the Safety Officers Field Manual.	
☐	☐	Are flag persons provided when operations or equipment on or near a highway expose workers to traffic hazards? Do flag persons and persons working in proximity to a road wear high visibility vests? Are persons exposed to highway vehicle traffic protected by signs in all directions warning of the presence of the flag persons and the work? Do signs and distances from the work zone conform to federal and local regulations?	



MOTOR VEHICLES
29 CFR 1926 Subpart O. EM 385-1-1, Section 18

YES	NO		COMMENT
☐	☐	Motor vehicle operators have a valid permit, license, or certification of ability for the equipment being operated.	
☐	☐	Inspection, maintenance, and repair is according to manufacturer's requirements by qualified persons.	
☐	☐	Vehicles are inspected on a scheduled maintenance program.	
☐	☐	Vehicles not in safe operating condition are removed from service until defects are corrected.	
☐	☐	Glass in windshields, windows, and doors is safety glass. Any cracked or broken glass is replaced.	
☐	☐	Seatbelts are installed and worn.	
☐	☐	The number of passengers in passenger-type vehicles does not exceed the number which can be seated.	
☐	☐	Trucks used to transport personnel have securely anchored seating, a rear end gate, and a guardrail.	
☐	☐	No person is permitted to ride with arms or legs outside of a vehicle body; in a standing position on the body; on running boards; seated on side fenders, cabs, cab shields, rear of the truck or on the load.	
☐	☐	ATV operators possess a valid state driver's license, have completed an ATV training course prior to operation of the vehicle, and wear appropriate protective equipment such as helmets, boots, and gloves.	

EXCAVATING AND TRENCHING
29 CFR 1926 Subpart P. EM 385-1-1, Section 25

YES	NO		COMMENT
☐	☐	Has the known or estimated location of utility installations such as sewer, telephone, fuel, electric, water lines, or any other underground installations that may be expected to be encountered during excavation been determined before excavation? Have utility locations been verified by designated state services according to state regulations? Has the client provided clearance where state jurisdiction doesn't apply?	
☐	☐	Have overhead utilities in excavation areas been identified and either de-energized, shielded or barricaded so excavating equipment will not come within 10 feet?	
☐	☐	Are inspections of the excavation, the adjacent areas, and protective systems made daily and as necessary by a competent person?	
☐	☐	Are Protective systems in place as prescribed by the competent person?	
☐	☐	Is material removed from excavations managed so it will not overwhelm the protective systems?	
☐	☐	Are barriers provided between excavations and walkways?	
☐	☐	Are excavations by roadways barricaded to warn vehicles of presence or to prevent them from falling in?	
☐	☐	Is there a means of exit from the excavation every 25 feet?	
☐	☐	Is air monitoring required? If yes, Is it performed?	



ELECTRICAL
29 CFR 1926 Subpart K. EM 385-1-1, Section 11

YES	NO		COMMENT
☐	☐	Are electrical installations made according to the National Electrical Code and applicable local codes?	
☐	☐	Qualified electricians make all connections and perform all work within 10 feet of live electric equipment.	
☐	☐	Location of underground, overhead, under floor, behind wall electrical lines is known and communicated. Lines are documented by qualified person as de-energized where necessary.	
☐	☐	Workers understand they must not work near live parts of electric circuits, unless they are qualified as required by OSHA or are protected by de-energizing and grounding the parts, guarding the parts by insulation, or other effective means?	
☐	☐	Employees who regularly work on or around energized electrical equipment or lines are instructed in the cardiopulmonary resuscitation (CPR) methods.	
☐	☐	Workers are prohibited from working alone on energized lines or equipment over 600 volts.	
☐	☐	Are Ground-fault circuit interrupters (GFCI's) or is ground fault circuit protection provided to protect employees from ground-fault hazards for all 115 – 120 Volt, 15 and 20 amp receptacle outlets which are not a part of the permanent wiring of a building or structure at construction sites?	
☐	☐	Circuit breakers are labeled.	
☐	☐	Circuit breaker and all cabinets with exposed electric conductors are kept tightly closed.	
☐	☐	Unused openings (including conduit knockouts) in electrical enclosures and fittings are closed with appropriate covers, plugs, or plates.	
☐	☐	Sufficient access and working space is provided and maintained about all electrical equipment to permit ready and safe operations and maintenance.	
☐	☐	Motors are located within sight of their controllers or controller disconnecting means are capable of being locked in the pen position or is a separate disconnecting means installed in the circuit within sight of the motor.	
☐	☐	Are visual inspections of extension cords and cord-and-plug-connected equipment conducted daily? Is equipment found damaged or defective tagged and removed from service, and not used until repaired?	
☐	☐	Wet Areas - Is portable lighting used in wet or conductive locations, such as tanks or boilers operated at no more than 12 volts and protected by GFCIs.	
☐	☐	Are electrical installations in hazardous areas to NEC?	
☐	☐	Metal ladders and tools including tape measures or fabric with metal thread are prohibited where contact with energized electrically parts is possible.	
☐	☐	All extension cords are the three-wire type, designed and rated for hard or extra hard usage?	
☐	☐	Worn or frayed electrical cords or cables are taken out of service. Fastening with staples, hanging from nails or suspending extension cords by wire is prohibited.	
☐	☐	Electric wire/flexible cord passing through work areas is protected from damage such as foot traffic, vehicles, sharp corners, projections and pinching? Flexible cords and cables passing through holes are protected by bushings or fittings?	
☐	☐	Before an employee or contractor performs any service or maintenance on a system where the unexpected energizing, start up, or release of kinetic or stored energy could occur and cause injury or damage, the system is to be isolated. Only authorized persons may apply and remove lockouts and tags.	
☐	☐	Contractors planning to use hazardous energy control procedures submit their hazardous energy control plan to the WESTON site safety officer or designee before implementing lockout/tagout procedures.	
☐	☐	There is a site specific hazardous energy control plan that clearly and specifically outlines the scope, purpose, authorization, rules and techniques to be used for the control of hazardous energy.	
☐	☐	Workers possess the knowledge and skills required for the safe application, usage, and removal of energy controls.	



WELDING AND CUTTING
29 CFR 1926 Subpart J. EM 385-1-1, Section 10

YES	NO		COMMENT
☐	☐	Prior to performing welding, cutting or any other heat or spark producing activity, an assessment of the area is made by a competent person to identify combustible materials and potential sources of flammable atmospheres.	
☐	☐	Welders, cutters and their supervisors are trained in the safe operation of their equipment, safe welding and cutting practices, hot work permit requirements, and fire protection.	
☐	☐	Welding and cutting equipment is inspected daily before use. Unsafe equipment is taken out of use, replaced, or repaired.	
☐	☐	Workers and the public are shielded from welding rays, flashes, sparks, molten metal, and slag.	
☐	☐	Employees performing welding, cutting, or heating are protected by PPE appropriate for the hazards (e.g., respiratory, vision and skin protection).	
☐	☐	Compatible fire extinguishing equipment is provided in the immediate vicinity of welding or cutting operations.	
☐	☐	Drums, tanks, or other containers and equipment which have contained hazardous materials shall be thoroughly cleaned before welding or cutting. Cleaning shall be performed in accordance with NFPA 327, <u>Cleaning or Safeguarding Small Tanks and Containers, ANSI/AWS F4.1, Recommended Safe Practices for the Preparation for Welding and Cutting of Containers That Have Held Hazardous Substances</u> , and applicable health and safety plan requirements.	

HAND AND POWER TOOL SAFETY
29 CFR 1926 Subpart I. EM 385-1-1, Section 13

YES	NO		COMMENT
☐	☐	Power tools are from a manufacturer listed by a nationally recognized testing laboratory for the specific application for which they are to be used.	
☐	☐	Hand & power tools are inspected, maintained, tested, and determined to be in safe operating condition before use.	
☐	☐	Tools found to be unsafe are not used, tagged and repaired or destroyed.	
☐	☐	Users of tools are trained in safe use.	
☐	☐	Electrical tools have cords and plug connections in good repair.	
☐	☐	Electrical tools are effectively grounded or approved double insulated.	
☐	☐	Reciprocating, rotating, and moving parts of equipment are guarded if they may be accessed by employees or they otherwise create a hazard.	
☐	☐	Safety clips/retainers are installed and maintained on pneumatic impact tool connections.	
☐	☐	Chain saws have an automatic chain brake or anti-kickback device.	
☐	☐	Pneumatic and hydraulic hoses and fittings are inspected regularly.	
☐	☐	Employees who operate powder actuated tools are trained and carry valid operator's cards.	
☐	☐	Powder activated tools are stored in individual locked containers, when not in use and are not loaded until ready to use.	
☐	☐	Powder actuated tools are inspected for obstructions or defects daily before use.	
☐	☐	Powder actuated tool operators have appropriate PPE.	



RIGGING
29 CFR 1926 Subpart H. EM 385-1-1, Section 15

YES	NO		COMMENT
☐	☐	Rigging equipment is inspected as specified by the manufacturer, by a qualified person, before use on each shift and as necessary to assure that it is safe.	
☐	☐	Defective equipment is removed from service.	
☐	☐	Rigging not in use is removed from the work area, properly stored, and maintained in good condition.	
☐	☐	Wire rope removed from service for defects is cut up or plainly marked as unfit for use as rigging.	
☐	☐	The number of saddle clips used to form eyes in wire rope conforms with Table H-20, are spaced evenly and the saddles are on the live side.	
☐	☐	Chain rigging has a tag clearly indicating load limits, is inspected before initial use, then weekly, and is of alloyed metal.	
☐	☐	Fiber rope rigging is not used if it is frozen or has been subject to acids or excessive heat.	
☐	☐	Slings and their fittings and fastenings are inspected before use on each shift and as needed during use.	
☐	☐	Drums, sheaves, and pulleys on rigging hardware are smooth and free of surface defects that can damage rigging.	

MATERIAL HANDLING, STORAGE, AND DISPOSAL
29 CFR 1926 Subpart H. EM 385-1-1, Section 14

YES	NO		COMMENT
☐	☐	Employees are trained in and use safe lifting techniques.	
☐	☐	Materials are not moved or suspended over workers unless positive precautions have been taken to protect workers.	
☐	☐	Conveyors are constructed, inspected, & maintained by qualified persons according to manufacturer's recommendations.	
☐	☐	All conveyors are to be equipped with emergency stopping devices.	
☐	☐	Hazardous exposed moving machine parts are guarded mechanically, electrically or by location.	
☐	☐	Controls are clearly marked and/or labeled to indicate the function controlled.	
☐	☐	Taglines are used for suspended loads where the movement may be hazardous to persons.	
☐	☐	Material in storage is protected from falling or collapse by effective stacking, blocking, cribbing, etc.	
☐	☐	Walkways and aisles are to be kept clear.	
☐	☐	Materials are not stored on scaffolds or runways in excess of normal placement or in excess of safe load limits.	
☐	☐	Work areas and means of access are maintained safe and orderly.	
☐	☐	Tools, materials, extension cords, hoses or debris do not cause tripping or other hazards.	
☐	☐	Storage and construction sites are kept free from the accumulation of combustible materials.	
☐	☐	Waste materials and rubbish are placed in containers or, if appropriate, in piles. Waste materials are disposed of in accord with applicable local, state, or federal requirements.	



**FLOATING PLANT AND MARINE ACTIVITIES
29 CFR 1926 Subpart O. EM 385-1-1 Section 19**

YES	NO		COMMENT
☐	☐	Floating plants that are regulated by the USCG have current inspections and certificates.	
☐	☐	Before any floating plant is brought to the job site and placed in service it is inspected and determined to be in safe operating condition	
☐	☐	Periodic inspections are made such that safe operating conditions are maintained. Strict compliance with EM 385-1-1, Section 19 is expected.	
☐	☐	Plans are in place for removing or securing the plant and evacuation of personnel endangered by severe weather and other marine emergencies such as; fire, flooding, man overboard, hazardous materials incidents, etc.	
☐	☐	Means of access are properly secured, guarded, and maintained free of slipping and tripping hazards.	
☐	☐	Dredging operations follow guidelines as established in EM 385-1-1, Section 19.D.	

**PRESSURIZED EQUIPMENT AND SYSTEMS
29 CFR 1926 Subparts I, F. EM 385-1-1, Section 20**

YES	NO		COMMENT
☐	☐	Pressurized equipment and systems are inspected before being placed into service.	
☐	☐	Pressurized equipment or systems found to be unsafe are tagged "Out of Service-Do Not Use".	
☐	☐	Systems and equipment are operated, inspected, and maintained by qualified, designated personnel.	
☐	☐	Safe clearance, lockout/tagout procedures are followed as appropriate during maintenance or repair.	
☐	☐	Air hose, pipes, fittings are pressure-rated for the activity. Defective hoses are removed from service.	
☐	☐	Hoses aren't laid over ladders, steps, scaffolds, or walkways in a manner that creates a tripping hazard.	
☐	☐	The use of compressed air for personal cleaning is prohibited. The use of compressed air for other cleaning is restricted to less than 30 psig.	
☐	☐	Compressed gas cylinders are stored in well-ventilated locations.	
☐	☐	Cylinders in storage are separated from flammable or combustible liquids and from easily ignitable materials by at least 40 feet or by a minimum five feet tall, ½ -hour fire resistive partition.	
☐	☐	Stored cylinders containing oxidizing gases are separated from fuel gas cylinders by at least 20 feet or by a minimum five feet tall, ½ -hour fire resistive partition.	
☐	☐	Cylinder valve caps are in place when cylinders are in storage, in transit, or a regulator is not in place.	
☐	☐	Compressed gas cylinders in service are secured in substantial fixed or portable racks or hand trucks.	
☐	☐	Oxygen cylinders and fittings are kept away from, and free from oil and grease.	
☐	☐	Cylinder Storage areas are posted with the names of the gases in storage and with signs indicating "No Smoking or Open Flame".	
☐	☐	Cylinders are to be stored such that mechanical and corrosion damage is avoided. Cylinders are not to be stored in areas required as an egress path.	
☐	☐	Cylinders may be stored in the open outdoors, however, they must be protected from the ground to prevent corrosion and must be protected from temperatures that may exceed 125 degrees F.	



WORK PLATFORMS/SCAFFOLDS
29 CFR 1926 Subparts L, M, N. EM 385-1-1 Sections 21, 22

YES	NO		COMMENT
☐	☐	Work platforms are erected, used, inspected, tested, maintained and repaired according to manufacturer's requirements.	
☐	☐	Construction, inspection, and disassembly of scaffolds is under the direction of a competent person.	
☐	☐	Workers on scaffolding have been trained by a qualified person.	
☐	☐	Scaffolds are erected on a firm and level surface and are square and plumb.	
☐	☐	Scaffolds are not loaded in excess of rated capacity.	
☐	☐	Working levels of work platforms are fully planked or decked.	
☐	☐	Planks are in good condition and free from obvious defects.	
☐	☐	Fabricated frame scaffolding four times higher than the base width is secured to building/structure according to manufacturer's instruction and/or OSHA requirements.	
☐	☐	Working platforms of scaffolding over ten feet in height have guard rails meeting OSHA specifications. Fall protection is suggested at four feet or greater.	
☐	☐	Scaffolding/work platforms are accessed by means of a properly secured ladder or equivalent. Built on ladders conform to scaffold ladder requirements. Climbing of braces is not allowed.	
☐	☐	Crane supported work platforms are designed and used in accordance with OSHA standards.	
☐	☐	Elevating work platforms are operated, inspected, and maintained according to the equipment operations manual.	
☐	☐	Employees working in aerial lifts remain firmly on the floor of the basket. Employees use fall protection while in an aerial lift basket.	



WALKING AND WORKING SURFACES AND STAIRS
29 CFR 1926 Subparts L, M, X. EM 385-1-1, Sections 21, 22, 24

YES	NO		COMMENT
☐	☐	Work areas are clean, sanitary, and orderly	
☐	☐	Work surfaces are kept dry or appropriate means are taken to assure the surfaces are slip-resistant	
☐	☐	Accumulations of combustible dust are routinely removed.	
☐	☐	Aisles and passageways are kept clear and marked as appropriate.	
☐	☐	There is safe clearance for walking in aisles where motorized or mechanical handling equipment is operating.	
☐	☐	Materials or equipment is stored in such a way that sharp projections will not interfere with the walkway.	
☐	☐	Changes of direction or elevation are readily identifiable.	
☐	☐	Aisles or walkways that pass near moving or operating machinery, welding operations or similar operations are arranged so employees will not be subjected to potential hazards.	
☐	☐	Standard guardrails are provided wherever aisle or walkway surfaces are elevated more than 30 inches above any adjacent floor or the ground and bridges provided where workers must cross over conveyors and similar hazards.	
☐	☐	There are standard stair rails or handrails on all stairways having four or more risers or with an elevation of 30 or more inches.	
☐	☐	Stairways are at least 22 inches wide. (General Industry Standard)	
☐	☐	Stairs angle no more than 50 and no less than 30 degrees, risers are uniform from top to bottom (plus or minus 1/4 inch) and are provided with a surface that renders them slip resistant.	
☐	☐	Stairway handrails are not less than 36 inches above the leading edge of stair treads and have at least 3 inches of clearance between the handrails and the wall or surface they are mounted on.	
☐	☐	Where doors or gates open directly on a stairway, there is a platform provided so the swing of the door does not reduce the width of the platform to less than 20 inches.	
☐	☐	Where stairs or stairways exit directly into any area where vehicles may be operated, there are adequate barriers and warnings provided to prevent employees stepping into the path of traffic.	
☐	☐	Signs are posted showing the load capacity of elevated storage areas.	
☐	☐	An appropriate means of access and egress is provided for surfaces with 19 or more inches of elevation change.	
		Material on elevated surfaces is minimized, with that necessary for immediate work requirements piled, stacked, or racked in a manner to prevent it from tipping, falling, collapsing, rolling, or spreading.	

FLOOR AND WALL HOLES AND OPENINGS
29 CFR 1926 Subpart M. EM 385-1-1, Section 24

YES	NO		COMMENT
☐	☐	Floor and roof openings that persons can walk into or fall through are guarded by a physical barrier or covered.	
☐	☐	Holes (defined as equal to or greater than 2 inches in least dimension) where person could trip must be covered/protected.	
☐	☐	Unprotected sides and edges on a walking/working surface six feet or more (note four feet in General Industry) are protected by guardrail system, safety net, or Personal Fall Arrest System (PFAS).	
☐	☐	Unused portions of service pits and pits not actually in use are either covered or protected by guardrails or equivalent.	
☐	☐	Coverings for holes or other openings must be constructed of sufficient strength to support any anticipated load, must be secured in place to prevent accidental removal or displacement, and must be marked indicating purpose (e.g., stenciled "Hole" or painted contrasting color to surroundings).	



LADDERS
29 CFR 1926 Subpart X. EM 385-1-1, Section 21

YES	NO		COMMENT
☐	☐	Portable ladders are used for their designed purpose only.	
☐	☐	Portable ladders are examined for defects prior to, and after use.	
☐	☐	Ladders found to be defective are clearly tagged to indicate "DO NOT USE" if repairable, or destroyed immediately if no repair is possible.	
☐	☐	Workers are trained in hazards associated with ladder use and how to inspect ladders.	
☐	☐	Ladders have secure footing provided by a combination of safety feet, top of ladder tie-offs and mud cills or a person holding the ladder to prevent slipping.	
☐	☐	The handrails of a straight ladder used to get from one level to another extend at least 36 inches above the landing.	
☐	☐	Ladders conform to construction criteria of ANSI Standards A-14.1 and A-14.2.	
☐	☐	Wooden ladders are not painted with an opaque covering such that signs of flaws, cracks, or drying are obscured.	
☐	☐	Fixed ladders are constructed and used according to OSHA Standards, 29 CFR 1910.27 and ANSI A-14.3.	
☐	☐	Rungs, cleats or steps, and side rails that may be used for handholds when climbing, offer adequate gripping surface and are free of splinters, splinters or burrs, and substances that could cause slipping.	
☐	☐	Fixed ladders of greater than 24 feet have cages or other approved fall protection devices. (Note General Industry is 20 feet).	
☐	☐	Where fall protection is provided by ladder safety systems (body belts or harnesses, lanyards and braking devices with safety lines or rails), systems meet the requirements of and are used in accordance with WESTON Fall Protection Standard Practices and are compatible with construction of the ladder system.	

DEMOLITION
29 CFR 1926 Subpart T. EM 385-1-1, Section 23

YES	NO		COMMENT
☐	☐	Prior to initiating demolition activities an engineering survey (by a competent person) and a demolition plan (by a competent person) is completed.	
☐	☐	All employees engaged in demolition activities are instructed in the demolition plan.	
☐	☐	It has been determined through the engineering survey and outlined in the plan, if any hazardous materials or conditions (e.g., asbestos, lead, utility connections, etc.) exist. Such hazards are controlled or eliminated before demolition is started.	
☐	☐	Continued inspections, by a competent person, are conducted to ensure safe employee working conditions.	



TREE MAINTENANCE AND REMOVAL
29 CFR 1910 Subpart R. EM 385-1-1, Section 31

YES	NO		COMMENT
☐	☐	Tree maintenance or removal is done is under the direction of a qualified person.	
☐	☐	Tree work, in the vicinity of charged electric lines, is by trained persons qualified to work with electricity and tree work. Appropriate distances are maintained for all workers who are not qualified.	
☐	☐	Equipment is inspected, maintained, repaired, and used in accordance with the manufacturer's directions.	
☐	☐	Prior to felling actions are planned to include clearing of the area to permit safe working conditions and escape.	
☐	☐	Employees must be trained in the safe operation of all equipment.	
☐	☐	All equipment and machinery is inspected and determined safe prior to use.	
☐	☐	Work is performed under requirements of FLD 43.	

BLASTING
29 CFR 1926 Subpart U. EM 385-1-1, Section 29

YES	NO		COMMENT
☐	☐	A blasting safety plan is developed prior to bringing explosives on-site.	
☐	☐	The transportation, handling, storage, and use of explosives, blasting agents, and blasting equipment must be directed and supervised by a person with proven experience and ability in blasting operations. Licensing of person is verified.	
☐	☐	Blasting operations in or adjacent to cofferdams, piers, underwater structures, buildings, structures, or other facilities must be carefully planned with full consideration to potential vibration and damage.	

HAZARDOUS, TOXIC, AND RADIOACTIVE WASTE AND UNDERGROUND STORAGE TANK (UST) ACTIVITIES
29 CFR 1926 Subpart D. EM 385-1-1, Section 28

YES	NO		COMMENT
☐	☐	All construction activities performed with known or potential exposure to hazardous waste are conducted in accordance with Hazardous Waste Operations and Emergency Response requirements.	



CONCRETE and MASONRY CONSTRUCTION
29 CFR 1926 Subpart Q. EM 385-1-1, Section 27

YES	NO		COMMENT
☐	☐	Construction loads are not placed on a concrete or masonry structure or portion of a concrete or masonry structure unless the employer determines, based on information from a person who is qualified in structural design, that the structure or portion of the structure is capable of supporting the loads.	
☐	☐	Employees are not permitted to work above or in positions exposed to protruding reinforcing steel or other impalement hazards unless provisions have been made to control the hazard.	
☐	☐	Sections of concrete conveyances and airlines under pressure are secured with wire rope (or equivalent material) in addition to the regular couplings or connections.	
☐	☐	Structural and reinforcing steel for walls, piers, columns, and similar vertical structures is supported and/or guyed to prevent overturning or collapse	
☐	☐	All form-work, shoring, and bracing is designed, fabricated, erected, supported, braced, and maintained so it will safely support all vertical and lateral loads that may be applied until the loads can be supported by the structure.	
☐	☐	Shoring equipment is inspected prior to erection to determine that it is specified in the shoring design. Any equipment found to be damaged is not used.	
☐	☐	Erected shoring equipment is inspected immediately prior to, during, and immediately after the placement of concrete. Any shoring equipment that is found to be damaged, displaced, or weakened is immediately reinforced or re-shored.	
☐	☐	Shoring, vertical slip forms and jacks conform with requirements of Section 27.B.08-13 of USACE EM 385-1-1.	
☐	☐	Forms and shores (except those on slab or grade and slip forms) are not removed until the individual responsible for forming and/or shoring determines that the concrete has gained sufficient strength to support its weight and all superimposed loads.	
☐	☐	Precast concrete members are adequately supported to prevent overturning or collapse until permanent connections are complete	
☐	☐	No one is permitted under pre-cast concrete members being lifted or tilted into position except employees required for the erection of those members.	
☐	☐	Lift slab operations are planned and designed by a registered engineer or architect.	
☐	☐	Hydraulic jacks used in lift slab construction have a safety device that causes the jacks to support the load in any position if the jack malfunctions	
☐	☐	No one is permitted under the slab during jacking operations.	
☐	☐	A limited access zone is established whenever a masonry wall is being constructed.	
☐	☐	Fall protection is provided to masonry workers exposed to falls of 6 feet or more.	



STEEL ERECTION
29 CFR 1926 Subpart R. EM 385-1-1, Section 27

YES	NO		COMMENT
☐	☐	Impact wrenches have a locking device for retaining the socket. Containers shall be provided for storing or carrying rivets, bolts, and drift pins, and secured against accidental displacement when aloft.	
☐	☐	Structural and reinforcing steel for walls, piers, columns, and similar vertical structures shall be guyed and supported to prevent collapse	
☐	☐	No loading is placed upon steel joists until all bridging is completely and permanently installed.	
☐	☐	Workers are provided fall protection whenever they are exposed to falls of 1.8 m (6 ft) or more (EM 385-1-1).	
☐	☐	Temporary flooring in skeleton steel erection conforms with Section 27.F of USACE 385-1-1	

ROOFING
29 CFR 1926 Subpart M. EM 385-1-1, Sections 21, 22, 24, 27

Yes	No		COMMENT
☐	☐	In the construction, maintenance, repair, and demolition, of roofs, fall protection systems is provided that will prevent personnel from slipping and falling from the roof and prevent personnel on lower levels from being struck by falling objects	
☐	☐	On all roofs greater than 4.8 m (16 ft) in height, a hoisting device, stairways, or progressive platforms are furnished for supplying materials and equipment.	
☐	☐	Roofing materials and accessories that could be moved by the wind, including metal roofing panels, that are on the roof and unattached are secured when wind speeds are greater than, or are anticipated to exceed, 10 mph.	
☐	☐	Level, guarded platforms are provided at the landing area on the roof.	
☐	☐	When their use is permitted, warning line systems comply with USACE Section 27.07 of EM 385-1-1.	
☐	☐	Workers involved in roof-edge materials handling or working in a storage area located on a roof with a slope $\geq$ to four vertical to twelve horizontal and with <u>edges 6 ft or more above</u> lower levels are protected by the use of a guardrail, safety net, or personal fall arrest system along all unprotected roof sides and edges of the area.	



ENVIRONMENTAL COMPLIANCE

Yes	No		Comments
☐	☐	Environmental Compliance and Waste Management Plan on file.	
☐	☐	Waste Determination Made.	
☐	☐	Manifest and/or Shipping Papers prepared and filed.	
☐	☐	Manifest Exception Reports Prepared, as necessary. Procedures to track manifests in place.	
☐	☐	State Annual and EPA Biennial Reporting Information Available.	
☐	☐	RCRA Personnel Training Records on file.	
☐	☐	CAA Permits on file.	
☐	☐	CWA Permits on file.	
☐	☐	RCRA Permits on file.	
☐	☐	State and/or Local Permits on file.	
☐	☐	RCRA Inspections conducted and Documentation on file.	
☐	☐	Transporter and TSD compliance information on file.	
☐	☐	Waste Accumulation Areas Managed Properly.	
☐	☐	Wetlands Areas Identified and Protected.	
☐	☐	Endangered, Threatened, or Special Concern Species or Areas Identified and Protective Methods Determined.	
☐	☐	Run-on and Runoff Concerns Identified and Managed.	
☐	☐	Adjacent Land Areas Protected as Necessary.	
☐	☐	Non-Hazardous Solid Wastes Managed Properly.	



MISCELLANEOUS REGULATORY and POLICY COMPLIANCE

Yes	No		Comments
☐	☐	Personnel Training Records for DOT Materials Handling on file.	
☐	☐	Noise Control Issues Addressed and Managed.	
☐	☐	Site Security Issues Identified and Managed.	
☐	☐	Known Historical, Archeological, and Cultural Resources Identified and Managed.	
☐	☐	WESTON EHS Analysis Checklist In Use.	
☐	☐	Safety Observation and Recognition Program in place.	
☐	☐	Weekly EHS Report Card System in place.	
☐	☐	Federal, State, and Local Required Postings in place.	
☐	☐	Site specific Lockout/Tagout Program is in place.	
☐	☐	Site-specific Confined Space Program is in place.	
☐	☐	Site Safety Officer filing system is in place and up to date.	



APPENDIX B

COMMUNITY AIR MONITORING PLAN (CAMP)



Community Air Monitoring Plan

Study Area – Pre-Design Investigation Work Plan

**Corning, NY
NYSDEC Project IS 851046**

January 9, 2018

Prepared for
**Corning Incorporated
Corning, New York**

Prepared by
**WESTON SOLUTIONS, INC.
West Chester, Pennsylvania 19380**

W.O. No. 02005.056.002



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LIST OF ACRONYMS

CAMP	Community Air Monitoring Plan
COPCs	constituents of potential concern
HASP	Health and Safety Plan
mg/m ³	milligrams per cubic meter
µg/m ³	micrograms per cubic meter
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PDI	Pre-Design Investigation
WESTON®	Weston Solutions, Inc.



1. INTRODUCTION

This Community Air Monitoring Plan (CAMP) has been prepared by Weston Solutions, Inc. (WESTON®) on behalf of Corning Incorporated to detail the dust control and air monitoring procedures to be performed during the execution of investigation activities at the Study Area located in Corning, New York, bound by the Chemung River to the south; Post Creek and Interstate 86 to the east and north; and the Guthrie Medical Center, the City of Corning Fire Department, and Centerway to the west (Study Area). This air monitoring plan will supplement the existing Health and Safety Plan (HASP).

As presented in the Study Area Pre-Design Investigation (PDI Work Plan), investigation activities planned to be conducted within the Study Area may include subsurface soil sampling. Air monitoring for dust particulates and dust control techniques will be performed during subsurface soil sampling activities.

2. METHODS

Perimeter air monitoring for dust particles will be conducted at two stations, one generally located upwind, and one generally located downwind of any intrusive characterization activity. In addition, due to the close proximity of athletic playing fields and schools, more stringent CAMP requirements will be necessary. When work areas are within 20 feet of these locations, the continuous monitoring locations for particulates must reflect the nearest potentially exposed individuals. The use of engineering controls such as dust barriers will be considered to prevent exposures related to the work activities and to control dust and odors. Where possible, consideration will be given to implementing the planned activities when potentially exposed populations are at a minimum (i.e. during hours when children are not likely to be present). Common-sense measures to keep dust and odors at a minimum around the work areas will also be implemented to ensure that the children are protected at all times. No visible dust will leave the work area.

As the location of investigation activities will change, the location of the monitoring point relative to the activities will be modified as needed. The monitoring location will be positioned to provide data representative of potential migration of dust in the direction of nearby receptors. The



perimeter monitoring equipment will be portable, which will allow the monitoring network to be adjusted if needed to adapt to changes in activities or meteorological conditions.

Particulate monitoring is the measurement of fine liquid or solid particles such as dust, smoke, mist, fumes or smog, in particle sizes less than 10 microns (PM_{10}), in the ambient air. During subsurface soil sampling and groundwater monitoring well installation, the generation of dust particles will be monitored. The equipment selected to monitor PM_{10} will be the Thermo Electron Corporation personal DataRAM (pDR), or equivalent. The pDR is a light-scattering monitor, designed for measuring airborne particulates such as aerosols and dusts. The units are portable and measure the concentration of airborne particulate matter (up to 10 μm in size) continuously and in real time, with results expressed in milligrams per cubic meter (mg/m^3), or 1,000 micrograms per cubic meter ($\mu g/m^3$). Particulate concentrations can be measured over the following ranges: 0.01 – 10 mg/m^3 (equivalent to 10 – 10,000 $\mu g/m^3$) and 0.1 – 100 mg/m^3 (equivalent to 100 – 100,000 $\mu g/m^3$). The pDR meets performance standard for a real-time particulate monitor according to the New York State Department of Environmental Conservation (NYSDEC) Technical Guidance for Site Investigation and Remediation; May 2010.

3. CALIBRATIONS

Calibration of instruments will be performed prior to the start of daily activities. Additional calibrations will be performed as needed or whenever maintenance is performed involving the functional elements of the unit. Calibration data will be documented in the field log book or on designated calibration log sheets.

4. DATA RECORDING

The data collected during the monitoring program will be used for real-time data display and notification to on-site personnel when the action levels are exceeded (action levels are discussed in Section 5). All ambient air monitoring data will be recorded in the site field logbook or designated field sheets and the results of the air monitoring will be communicated to the NYSDEC and New York State Department of Health (NYSDOH) on scheduled basis (i.e. daily for levels which require actions, weekly for routine monitoring data).



5. ACTION LEVELS

The action level established herein will be used as an indicator that potential excessive migration of dust particles may be occurring during the characterization activities. Monitored ambient air concentrations above the action level will result in actions being taken to more stringently control fugitive emissions or trigger quantitative sampling.

The NYSDEC recommended action level for fugitive dust is $100 \mu\text{g}/\text{m}^3$ greater than background (measured at the upwind location) for a 15 minute period. At this concentration, work may continue with dust suppression techniques provided that no visible dust is migrating from the working area, and the downwind particulate levels do not exceed $150 \mu\text{g}/\text{m}^3$ greater than background (measured at the upwind location). If the downwind particulate levels exceed $150 \mu\text{g}/\text{m}^3$ greater than background (measured at the upwind location), work will stop and dust suppression techniques will be re-evaluated.

If the perimeter monitors detect concentrations above the $100 \mu\text{g}/\text{m}^3$ action level, Site supervisory personnel will be notified. Notifications will be sent to the WESTON Site Manager and the Site Health and Safety Officer. Upon receiving the notification message, the supervisor will assess the situation and initiate appropriate administrative and/or engineering controls to mitigate the migration of dust particles.



APPENDIX C

QUALITY ASSURANCE PROJECT PLAN (QAPP)



Quality Assurance Project Plan

Study Area

**Corning, NY
NYSDEC Project ID 851046**

January 9, 2018

Prepared for

**Corning Incorporated
Corning, New York**

Prepared by

**WESTON SOLUTIONS, INC.
West Chester, Pennsylvania 19380**

W.O. No. 02005.056.002



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APPENDICES

ATTACHMENT A	TestAmerica Quality Assurance Manual and Standard Operating Procedures

LIST OF ACRONYMS

ASP	Analytical Services Protocol
ASTM	American Society of Testing and Materials
bgs	below ground surface
CAMP	Community Health and Safety Plan
COPC	constituents of potential concern
CPR	cardiopulmonary resuscitation
DAR	data applicability report
DQO	data quality objectives
DUSR	data usability summary report
EDD	electronic data deliverable
ELAP	Environmental Laboratory Approval Program
GPR	ground penetrating radar
HASP	Health and Safety Plan
ID	sample identification
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LCSRM	laboratory control sample reference material
MCAWW	Methods for Chemical Analyses of Waters and Wastes
MDL	method detection limits
MS	matrix spike
MSD	matrix spike duplicate
ND	non detect
NYSDEC	New York State Department of Environmental Conservation
NYSDEC	New York State Department of Health
OU	operation unit
OSHA	Occupational Safety and Health Administrations
PDI	pre-design Investigation
PID	Photoionization Detector
PPE	personal protective equipment
QA	quality assurance



LIST OF ACRONYMS (Continued)

QAM	TestAmerica Quality Assurance Manual
QAO	quality assurance objectives
QAPP	Quality Assurance Project Plan
QC	quality control
RPD	relative percent difference
SOP	standard operating procedure
SOW	scope of work
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
VOC	volatile organic compounds
WESTON®	Weston Solutions, Inc.



1. INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared by Weston Solutions, Inc. (WESTON®) on behalf of Corning Incorporated to detail the quality assurance/quality control (QA/QC) procedures for conducting field activities in the Study Area located in Corning, New York as illustrated on Figure 1-1. In general the Study Area is bound by the Chemung River to the south; Post Creek and Interstate 86 to the east and north; and the Guthrie Medical Center, the City of Corning Fire Department, and Centerway to the west. The Study Area is separated into five operable units (OUs) based on location and land use, to assist in advancing properties through the remediation process. The five OUs in the Study Area are identified as follows: the Residential Area (OU1), the Residential Area at the Eastern End of Corning Boulevard (OU2), School/Community Use Areas (OU3), Flood Control Areas (OU4), and the Residential Expansion Area (OU5). The Study Area and OUs are depicted on Figure 1-2.

The characterization activities are detailed in the Study Area Pre-Design Investigation Work Plan, (PDI Work Plan) and approved by the New York State Department of Environmental Conservation (NYSDEC).

1.1 PROJECT SCOPE AND GOALS

The purpose of the PDI Work Plan is to gather additional data to assist in defining the extent of Subject Material (defined in Section 1.1 of the PDI Work Plan) encountered within OU1, OU2 and OU5. This additional data will help determine the limits of remediation under the selected remediation alternative of soil excavation up to 2 feet below ground surface (ft bgs) and a soil cover.

The specific objectives of the PDI Work Plan are as follows:



1. Perform additional sampling, as needed, to place on all residential properties in OU1 and OU5 at least four surface soil samples and two soil borings¹.
2. Perform additional sampling to delineate Subject Material.
3. Perform additional sampling to investigate fill material containing ash, brick and/or glass where analytical results collected to date do not indicate that concentrations of constituents are greater than the New York State Department of Environmental Conservation (NYSDEC) Soil Cleanup Objectives (SCOs; New York Codes, Rules and Regulations (NYCRR(Subpart 37506)².

Additional details, including figures and tables describing the proposed sampling locations and number of samples are included in the PDI Work Plan. Due to the confidential nature of sample collection on privately-owned residential properties, no sampling location maps will be included in the PDI Work Plan. Sample locations are described in tabular form in Section 4.0 of the PDI Work Plan.

1.2 PROJECT DATA QUALITY AND OBJECTIVES

This QAPP documents the QA/QC measures that will be followed during the implementation of PDI Work Plan activities and any follow-up activities that may be conducted (if required). The objective of the data collection is to support the characterization activities within OU1, OU2 and OU5.

The QAPP provides a description of the analytical, field, and reporting procedures that may be used by WESTON and its subcontractors within the Study Area for the following activities:

- Soil boring installation;
- Soil sampling;
- Well installation;
- Monitoring well sampling;

¹ With the exception of a 66 properties in OU1 which have been included in the expedited sampling plan per the request of NYSDEC.

² NYSDEC residential SCOs are applicable to the residential properties and NYSDEC restricted-residential SCOs are applicable to the City of Corning rights-of-way.



- Well abandonment;
- Test pitting;
- Laboratory analysis; and
- Report preparation.

The purpose of the QA/QC program is to produce analytical measurement data of known quality that satisfy the project data quality objectives (DQOs). DQOs are data quality planning and evaluation tools for sampling and analysis activities. A consistent and comprehensive approach for developing and using these tools is necessary to ensure that enough data are produced and data are of sufficient quantity to make decisions for the project. The DQO process is described in the subsequent subsection.

1.3 DATA QUALITY OBJECTIVES

The DQO process and quality assurance objectives for program planning are presented in this section. The procedures of the overall QA/QC have been developed to ensure that the analytical data collected through implementation of the PDI Work Plan are of known and acceptable level of quality.

Primary DQOs will include the following:

- Complete the Study Area pre-design investigation activities to adequately confirm the presence/absence of Subject Material; and
- Provide additional information to fully characterize potential migration pathways.

To achieve the DQOs, QA measures will be implemented throughout the project to ensure that the data meet known and suitable data quality criteria such as selectivity, precision, accuracy/bias, representativeness, comparability, and completeness. The sampling data will be quality-controlled through the collection of field QC samples and the calibration of field and laboratory equipment. In addition, replicate samples will be collected and submitted as part of the QA program. Implementation of QA/QC measures to achieve the DQOs will limit the chance of generating inadequate or incomplete data.



The DQOs will be accomplished by ensuring that the following analytical objectives are met. These analytical objectives will include the following:

- To prepare and analyze samples using standard methods; and
- To obtain usable and defensible analytical results.

Quality assurance objectives (QAOs) are the detailed QC specification for selectivity, precision, accuracy, representativeness, comparability, and completeness. In regards to measurements of data quality, the QA/QC program will include the following QAOs:

- Provide a mechanism for the ongoing control and evaluation of measurement data quality; and
- Provide measures of data quality in terms of selectivity, precision, accuracy, completeness, representativeness, and comparability to assess whether the data meet the project objectives and can be used for their intended purpose.

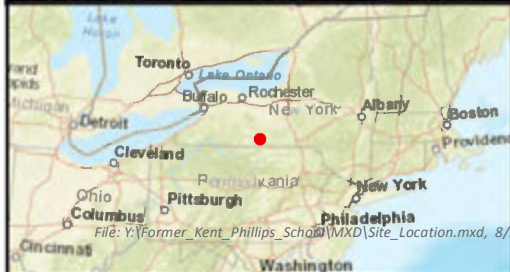
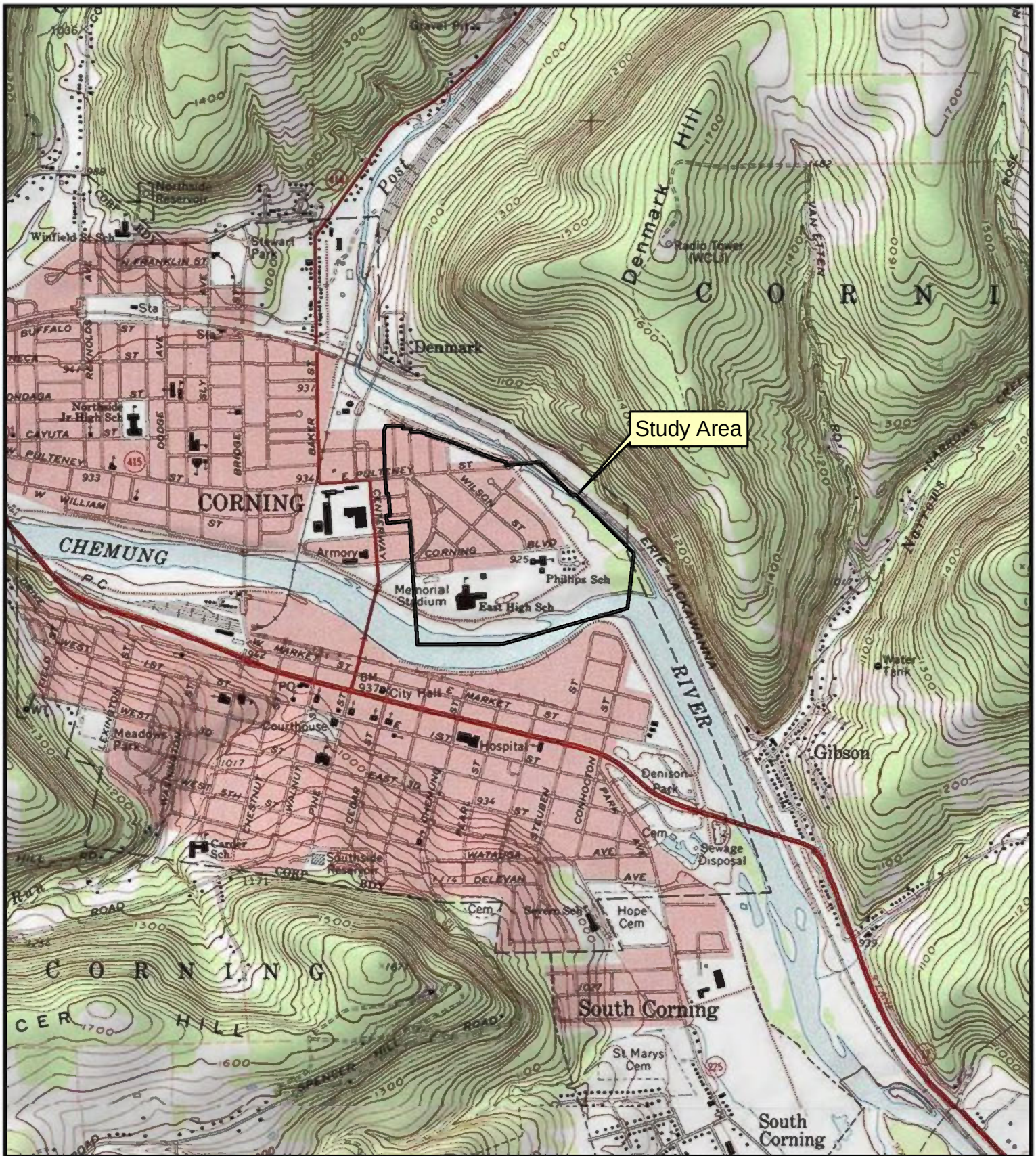
The primary application of analytical results will be to generate sufficient information to determine the presence or absence of Constituents of Potential Concern (COPCs) within OU1, OU2 and OU5 and/or the nature and extent of COPCs. The project data manager will track data from collection of samples through login at the laboratory to delivery by technical report and electronic data deliverable (EDD), oversee necessary validation, data usability summary report preparation (DUSR) and/or data applicability report preparation (DAR), and coordinate laboratory corrective actions.

The following sections discuss the steps to be taken to ensure the quality of data acquired. The representativeness of the measurement data is a function of the sampling strategy and will be achieved by following the procedures in the PDI Work Plan. The quality of the analytical results is a function of the analytical system and will be achieved by using standard methods and the QC practices discussed in this section. The basis for assessing selectivity, precision, accuracy, representativeness, comparability, and completeness is discussed in TestAmerica's QA Manuals (QAM) found in Attachment A.



1.4 PROJECT SCHEDULE

The schedule for project activities is presented in Section 5.1 of the PDI Work Plan.



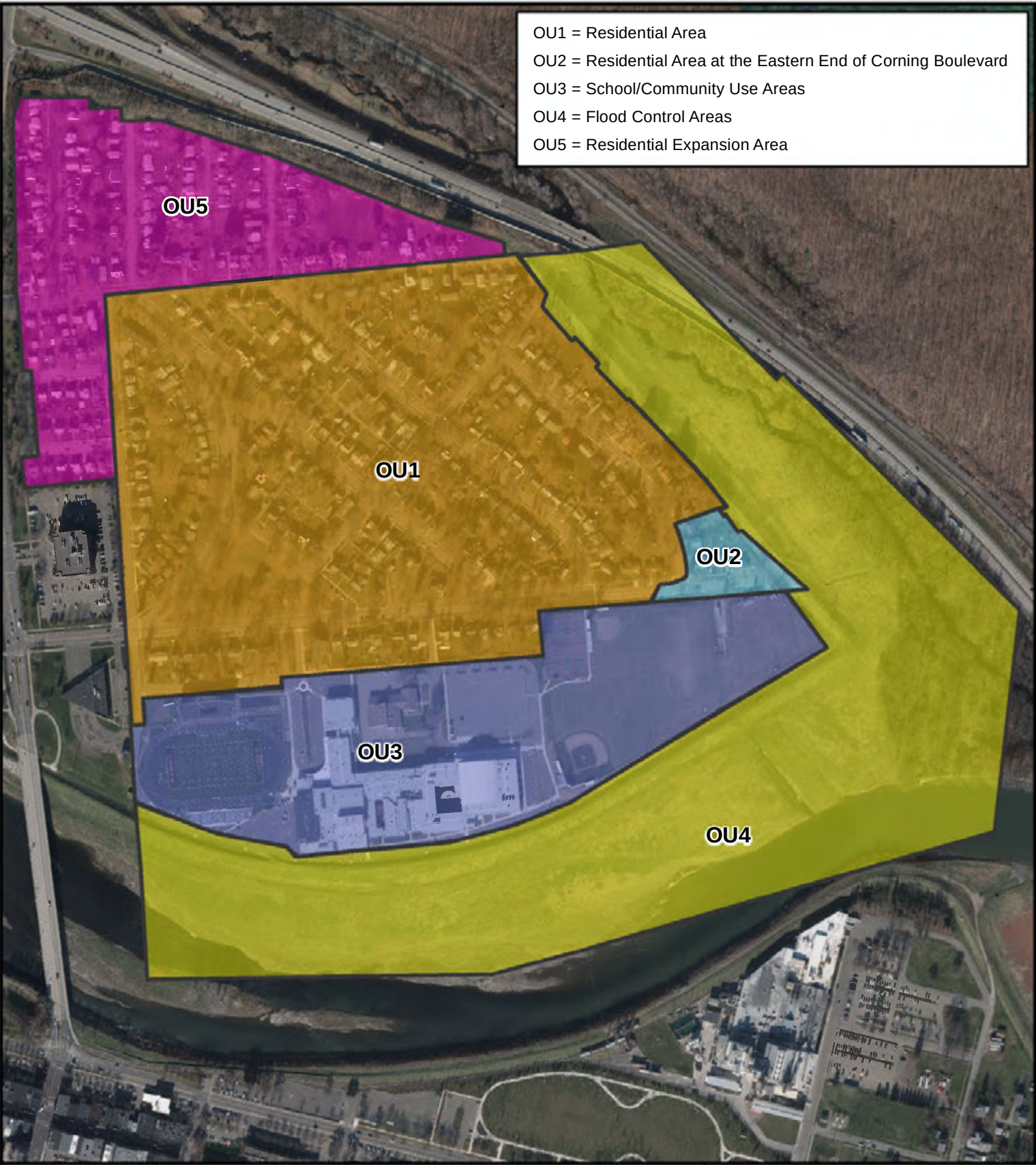
2

WESTON
SOLUTIONS

0 1,000 2,000
Feet

USGS 7.5 Minute Quadrangle Corning NY, 1971

Figure 1-1
Location of Study Area
Corning, NY



OU1 = Residential Area
OU2 = Residential Area at the Eastern End of Corning Boulevard
OU3 = School/Community Use Areas
OU4 = Flood Control Areas
OU5 = Residential Expansion Area

Legend Operable Units - OU1 - OU2 - OU3 - OU4 - OU5	2 WESTON SOLUTIONS 0 250 500 Feet Base Imagery: Robinson Aerial Imagery, Dec 2015 Coordinate System: NAD 1983 State Plane New York Central Feet Datum: NAD83. Units: Feet	Figure 1-2 Study Area Operable Units Corning, NY



2. PROJECT ORGANIZATION AND RESPONSIBILITIES

A general description of the organization and the responsibilities of key individuals for the project teams are provided in this section. This QAPP covers the work of Corning Incorporated, WESTON, and subcontractors. Responsibilities and authority may vary among subcontractors. The following sections give brief descriptions of the primary staff and the responsibilities of the management, QA/QC, and primary task leadership for the field and laboratory tasks. Project activities will be performed within the framework of the organization and functions described in this section.

The organization for the project is designed to provide clear lines of responsibility and authority. This control structure provides for the following:

- Identifying lines of communication and coordination;
- Monitoring project schedules and performance;
- Managing key technical resources;
- Coordinating support functions such as laboratory analysis and data management; and
- Rectifying deficiencies.

QA personnel will have sufficient authority, organizational freedom, and ability to act as follows:

- Identify QA problems.
- Initiate, recommend, or provide solutions to QA problems through designated channels.
- Ensure that program activities, including processing information deliverables, and installation or use of equipment, are reviewed in accordance with QA objectives.
- Ensure that deficiencies/non-conformances are corrected.
- Ensure that further processing, delivery, or use of data is controlled until the proper disposition of a nonconformance, deficiency, or unsatisfactory condition has occurred.



The organizational structure will be reviewed and updated periodically by the WESTON Project Manager. Any necessary staff changes will be filled with qualified personnel and communicated to the Corning Incorporated Project Manager.

2.1 CORNING INCORPORATED COMPANY PERSONNEL

Mr. Michael Ford, will serve as the Corning Incorporated Project Manager for the project. Mr. Ford is responsible for primary contact with NYSDEC and for oversight of the project. Mr. Ford's responsibilities include defining project objectives, allocating resources, determining the chain-of-command, and evaluating the project outcome.

Michael Ford
Corning Incorporated
HP-ME-03-83
Corning, NY 14831

2.2 WESTON PERSONNEL

2.2.1 Project Manager

Mr. John Sontag, Jr. will serve as the WESTON Project Manager for the project. Mr. Sontag will be responsible for day-to-day activities on the project and planning, coordinating, integrating, monitoring, and managing project activities, including the activities of subcontractors to WESTON. Mr. Sontag will also be responsible for the identification and ultimate resolution of technical problems and the technical coordination of the field efforts, and subsequent data assessment.

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(610) 701-3401 (fax)
john.sontag@westonsolutions.com



2.2.2 Technical Advisors/Quality Assurance Coordinator

Mr. Michael H. Corbin, P.E., and Mr. Thomas A. Drew, P.G., will serve as the WESTON Technical Advisors/Quality Assurance Coordinators. Messrs. Corbin and Drew will be responsible for reviewing the information generated during the program. Messrs. Corbin and Drew will have the authority to impose proper procedures or to halt an operation. Their duties include QA review and approval of sampling activities, field documentation, and technical data as well as conducting QA audits if needed.

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2.2.3 Field Team Project Manager/Health and Safety Officer

Ms. Rachel McLoughlin, Ms. Audra Balson, Ms. Devon Hollenden, and Mr. John Sontag will lead the Field Team, serve as Health and Safety Officers, and be responsible for oversight of activities in the field. She/He will be responsible for ensuring that procedures for the field activities related to soil sampling and groundwater sampling are executed in accordance with the PDI Work Plan and are documented according to the procedures presented in this QAPP. The Health and Safety Officer will be responsible for: (1) having an up-to-date Health and Safety Plan (HASP) and Community Air Monitoring Plan (CAMP) in place, (2) ensuring that all WESTON and subcontractor personnel adhere to the HASP and CAMP protocols, (3) training personnel involved in health and safety procedures, (4) maintaining control and exercising proper response in emergencies, and (5) keeping a logbook of activities.



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2.2.4 Data Manager

Ms. Amanda McGurk of WESTON will be responsible for managing the analytical data generated from the project activities.

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amanda.mcgurk@westonsolutions.com

2.3 LABORATORY STAFFING

Analytical work required during the project activities will be performed by TestAmerica Laboratories, Inc. (TestAmerica). The primary TestAmerica laboratory being located in Buffalo, NY. TestAmerica Buffalo may subcontract work to the Edison, NJ laboratory as required to maintain project timelines. These TestAmerica Laboratories are NYSDOH Environmental Laboratory Approval Program (ELAP)-certified laboratories (TestAmerica Buffalo certification #10026 and TestAmerica Edison certification #11452). If for any reason another laboratory is needed during the project activities, it will be required to comply with the requirements presented in this QAPP.



The TestAmerica QAMs for each laboratory are included as Attachment A of this QAPP. The laboratory QAMs include detailed explanations of the staff organization and QA system, as well as personnel responsibilities, qualifications, and training.

It is the individual responsibility of analysts and technicians to perform their assigned tasks according to this QAPP, applicable standard operating procedures (SOPs) and the PDI Work Plan for the project. This includes responsibility for performing QC analyses as specified in the method SOP and for entering the QC data in the appropriate logbook, electronic database, or method control file system. The analyst will report out-of-control results to the Laboratory Quality Manager and will indicate corrective action for out-of-control events.

2.3.1 Laboratory Personnel and Responsibilities

Ms. Judy Stone will serve as TestAmerica's Project Manager. The Project Manager is accountable for the oversight of all laboratory functions and operations, including coordination with/between WESTON and the Laboratory Quality Manager.

Mr. Brad Prinzi will serve as TestAmerica's Laboratory Quality Manager. The Quality Manager's responsibilities include the oversight of the laboratory's Quality Systems and ensuring that all tasks performed by the laboratory and TestAmerica field personnel are conducted in compliance with state, federal and industry standards, as well as the requirements of this QAPP.

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Amherst, New York 14228
484-685-0868

Brad Prinzi – Laboratory Quality Manager
TestAmerica Buffalo
10 Hazelwood Drive
Amherst, New York 14228
716-504-9800

2.3.2 Subcontractors

If subcontractors are required, the WESTON Project Manager will coordinate with the WESTON Subcontractor Administrator on developing the scope of work (SOW) to be performed by the subcontractors. The Field Team Manager will direct the subcontractors in the field in accordance with their specific SOW.



2.4 TRAINING AND CERTIFICATION

2.4.1 Field Staff Training and Certification

Information pertaining to project-specific training and certification can be found in the HASP prepared for the sampling activity, including;

- medical monitoring,
- Occupational Safety and Health Administrations (OSHA),
- first aid/cardiopulmonary resuscitation (CPR); and
- equipment operation, and associated records and documentation.

Training records for field staff, including subcontractors, will be available to the WESTON Project Manager.



3. FIELD SAMPLING PROCEDURES

This section describes the components of the sampling procedures that will be performed at the Study Area. The matrix, parameters and initial number of samples for characterization activities are presented in the sample summary table included in the PDI Work Plan (see Table 4-1 in the PDI Work Plan). Sampling locations, rationale, and analytical methods, as well as the sampling and decontamination procedures, for this project are discussed in detail in the PDI Work Plan and attached SOPs.

Prior to the sampling activities, the Field Team Manager will ensure that the field personnel understand the purpose, objectives and scope of the event. Topics of review and discussion with the team may include schedules, responsibilities, sampling locations, types of samples to be collected (both field samples and QC samples), number of samples and sample volumes to be collected, sample identification numbering schemes, preservation requirements, parameter(s) to be analyzed, sampling procedures, equipment decontamination procedures, and chain-of-custody requirements. The Field Team Manager will ensure that field personnel also have access to a copy of the PDI Work Plan including the SOPs. Field activities must be conducted in accordance with the health and safety procedures described in the HASP.

3.1 PRE-SAMPLING PROCEDURES

Sampling equipment (i.e., drill rigs and supporting equipment, hand augers, bailers, pumps and trowels) will be decontaminated prior to arrival or cleaned and decontaminated in accordance with the SOP (Appendix D of the PDI Work Plan). In accordance with the PDI Work Plan, dedicated disposable sampling equipment may also be used.

3.2 DRILLING PROCEDURES

Criteria for selecting soil boring(s), monitoring well(s), and soil sampling locations (i.e., drilling locations) are based on the specific objectives for each study area, as described in Section 4 of the PDI Work Plan. As described in Section 4, final selection of drilling locations will depend on securing all necessary clearances, written agreement for access, permits and approvals. If



necessary, electrical cable and pipe locator instruments will be used with underground utility maps, magnetometer readings, and ground penetrating radar (GPR) to determine if utilities underlie the drilling location.

Cores to be visually logged and samples to be collected for physical or chemical analysis will be collected and handled according to the procedures described in the PDI Work Plan. Field screening instrument calibrations will be conducted according to the procedures present in this QAPP.

3.3 SUBSURFACE SOIL SAMPLING PROCEDURES

Soil samples will be collected in the Study Area in accordance with the PDI Work Plan. Soil borings will be advanced via Geoprobe® or hollow-stem auger and shallow surface soil samples may be collected using a hand auger or scoop/trowel. Additional details regarding the locations of the samples are described in the PDI Work Plan (see Section 4) and in PDI Work Plan addendums.

The soil sampling procedure is described in the PDI Work Plan and contains of the following elements:

- Locations will be cleared by an underground utility survey (as needed).
- Soils will be visually logged and screened with photoionization detector (PID).
- Specific sampling intervals will be documented in the project field notebook and/or designated field sheets.
- Soil samples will be identified by location, sample type, sample location, QC type, and depth/location.
- Samples will be placed in an ice-filled cooler for shipment to the laboratory (as needed) depending on the laboratory method requirements.

The potential list for analysis of soil samples, including the soil sample container volume, type, hold times, and associated preservation method are summarized in Table 3-1. Additional information regarding the analytical methods is specified in the TestAmerica QAM.



3.4 GROUNDWATER SAMPLING PROCEDURES

The PDI Work Plan does not include a groundwater investigation program. If required, groundwater investigations will likely involve the installation of groundwater monitoring wells and potential sampling of existing wells (dependent upon access agreements with the well owners).

A groundwater sampling procedure would contain the following elements:

- Locations will be cleared by a utility survey (as needed).
- Groundwater wells will be installed approximately 10 feet below the water table.
- Total well depth measurements and groundwater level measurements will be recorded.
- Groundwater wells will be purged and sampled in accordance with the SOP.
- Samples will be identified by location, sample type, sample location, and QC type.
- Samples will be placed in an ice-filled cooler for shipment to the laboratory (as needed) depending on the laboratory method requirements.

The potential list of analysis for groundwater samples, including the sample container volume, type, hold times, and associated preservation method are summarized in Table 3-2. Additional information regarding the analytical methods is specified in the TestAmerica QAMs.

3.5 FIELD QUALITY CONTROL SAMPLES

QC samples will be collected and analyzed as stated in the following subsections. The frequency of sample collection will be as specified in the following subsections and in accordance with the PDI Work Plan (see Table 4-1 in the PDI Work Plan).

3.5.1 Equipment Rinsate Blanks

Analyses of equipment rinsate blanks will be used to assess the effectiveness of field equipment decontamination procedures in preventing cross-contamination between samples. De-ionized or distilled water will be poured into/through/over clean (decontaminated) sampling equipment used in the collection of investigative samples, and then collected into prepared sample bottles. The



rinsate blank will then be shipped with the environmental samples collected from the same parameter group. For each matrix, a rinsate blank will be collected and analyzed for every 20 samples (or less) collected. The rinsate blanks will be analyzed for the same parameters as the investigative samples. Rinsate blanks will not be collected when precleaned or dedicated equipment is used for sampling.

3.5.2 Trip Blanks

Trip blanks are volatile organic sample containers prepared in the laboratory using analyte-free water. The trip blanks will be included with samples to be analyzed for volatile organic compounds (VOCs) to assess the contamination of sample containers during transport, during sample collection, and during transport to the laboratory. Trip blank containers will be the same type of sample container as those used for the VOC samples. One trip blank sample will be included for each cooler of samples containing samples collected for analysis of VOCs. At no time after their preparation will the trip blanks be opened prior to reaching the laboratory. The trip blank will stay with the cooler until it is received at the laboratory. The trip blanks will be analyzed for VOCs, as appropriate.

3.5.3 Field Duplicate Samples

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate soil samples will be collected from the same sampling interval, where practical. Duplicate sample results will be used to assess precision, including variability associated with both the laboratory analysis and the sample collection process. For soil samples, they also provide a measure of the heterogeneity of the soil matrix. Duplicate samples will be collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation and analysis. One duplicate sample will be collected for every 20 samples. If fewer than 20 samples are collected, one duplicate will still be collected. These duplicates will be analyzed for the same sample parameters specified for the original sample. Duplicate water samples for VOC analysis will not be alternately split among containers, but will be directly poured into the appropriate containers until filled (i.e., grab sample). Duplicate soil



samples for non-VOC parameters will be collected from the homogenized sample for which the primary sample is collected. Duplicate soil sample for VOC parameters will be a grab sample.

3.5.4 Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

MS/MSDs are samples in which known amounts of compounds are added in the laboratory before extraction and analysis. Two aliquots of the sample will be spiked for the duplicate analysis. The results of the duplicate spiked samples will be used to measure the percent recovery of each spiked compound and compare the recovery between samples, which will provide estimates of the accuracy and precision of the method. The solution of target analytes in MSs for organic analyses is based on SW-846 methods and does not include all target analytes, but is rather a representative subset. When reviewed in conjunction with other QC data, MS/MSDs data may indicate the need for reanalysis using a more appropriate method. For each matrix type, at least one spiked set of MS/MSDs will be analyzed for each batch of samples for every 20 (or less) samples received. The MS/MSD portion of the sample will be collected in a separate bottle for the routine sample to provide sufficient sample volume and to allow for the assessment of unspiked results for field precision.

3.6 SAMPLE HANDLING

Sampling and preservation procedures will be as mandated by each respective method. In order to preserve the integrity of the sample before it is analyzed, proper sample containment, and shipping and chain-of-custody procedures will be followed.

3.6.1 Sample Custody

This section contains a basic discussion of sampling custody practices. The QC practices contained in this section are intended to address potential problems with labeling errors, transcription errors, and preservation errors. Overall, the QC checks included in this section are the mechanisms that detect and correct errors.

An overriding consideration for environmental data is the ability to demonstrate that samples were obtained from the locations stated and that they reached the laboratory without alteration. The sample custody procedures provide a mechanism for documentation of information related to sample



collection and handling to achieve this objective. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal will be documented to accomplish this goal. Documentation will be accomplished through a chain-of-custody that records each sample and the individuals responsible for sample collection, shipment, and receipt. A standard chain-of-custody form has been provided by TestAmerica (see Figure 3-1).

All samples that are collected will be accompanied by a chain-of-custody record. Information to be recorded on the laboratory supplied chain-of-custody includes:

- Project name and number.
- Initials of sampler.
- Sample number, location, date and time collected, and sample type.
- Analyses requested.
- Any special instructions and/or sample hazards.
- Signature of sampler in the designated blocks, indicating date, time, and company.
- Condition of the sample upon receipt as reported by the analytical laboratory.

The purpose of sample custody procedures is to document the history of sample containers and samples from the time of sample collection through shipment and analysis. An item is considered to be in one's custody if one or more of the following conditions apply:

- It is in a person or company's actual possession.
- It is in view after being in physical possession.
- It is secured so that no one can tamper with it after having been in physical custody.

The following chain-of-custody procedures will be followed for samples submitted to the laboratory for chemical or physical properties analysis:

- Each individual field sampler is responsible for the care and custody of samples he or she collects until the samples are properly transferred to temporary storage or for shipping.



- A chain-of-custody record will be completed by the sampler for samples collected and submitted to the laboratory.
- Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time, will be documented. If shipment is required, shipment records may be used to document receipt/relinquishment of the samples.
- The laboratory will record the condition of the sample containers upon receipt.
- Changes or corrections to the information documented by the chain-of-custody form (including, but not limited to, field sample ID or requested analyses) must be changed and initialed by the person requesting the change.
- A copy of the chain-of-custody form and any documented changes to the original will be returned from the laboratory as part of the final analytical report to the Project Manager. This record will be used to document sample custody transfer from the sampler to the laboratory and will become a permanent part of the project file.

As an essential part of project management, WESTON has established sample control procedures to ensure sample integrity. Sample containers and samples will be maintained throughout the project activities.

3.6.2 Sample Identification

A unique sample code, known as a field sample identifier or sample identification (ID), will be assigned to each sample collected. The field collection system will be set up to allow the Field Team Manager, or designated sampling coordinator, to generate field sample identifiers prior to sample collection, if sufficient information is known (i.e., number of sampling locations and depths). Each unique identifier will be printed on the sample jar label, along with the date, and time of sample collection. In addition, numeric or alphabetic value will be assigned to the type of sample (i.e., primary sample, field duplicate, and rinse blank) to distinguish samples that will be used for QC purposes.

Field sample identifiers will be generated so that there is no duplication and recorded on the chain-of-custody. The format that will be followed for the field sample identifiers during the characterization activities at the Study Area is presented in Table 3-2.



3.6.3 Sample Labels

Each sample collected will be labeled with the assigned sample identification, which will be on the label attached to the sample container. Additional information recorded on the sample label includes where it was collected, when it was collected, the analysis required, preservative (if any) and identification of the sample(s).

Chain-of-custody records will be numbered to facilitate tracking of the shipment of individual samples. After the sample identification information is entered in the field logbook or designated sampling sheets, it will be entered on the chain-of-custody form and shipped with the samples.

3.6.4 Shipping Procedures

The objective of sample handling procedures is to ensure that samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. It is anticipated that samples will be delivered to TestAmerica Laboratory via a WESTON employee or a laboratory courier service, however samples may be shipped via Fed-Ex or other third-party carrier as needed.

3.7 LABORATORY OPERATIONS

TestAmerica Laboratories will follow the most recent versions of QAMs and SOPs (see Attachment A of this QAPP for current versions) for laboratory custody procedures, handling, identification, control, and chain-of-custody procedures and to maintain the validity of the samples.

3.7.1 Sample Receipt

Upon receipt of the samples at the laboratory, a sample custodian, familiar with custody requirements and the potential hazards of handling environmental samples will receive the samples. In addition, the sample custodian will also be responsible for documenting sample receipt, storage before and after sample analysis, and the proper disposal of samples. Upon sample receipt, the sample custodian will do the following:

- Sign the chain-of-custody and place it in the project file.



- Inspect samples for condition upon receipt, type and status of refrigerant, hold times, and turnaround time requirements.
- Log in samples and assign each with a unique sample number.
- Assign each sample a unique barcode label and place in proper storage area until the samples are ready to be prepared/analyzed.

3.7.2 Sample Storage

Samples will be stored in the proper environment as directed by the Laboratory Project Manager as described in Attachment A of this QAPP. To prevent mix-ups and cross-contamination, samples will be stored in areas as designated in the applicable SOP (provided in Attachment A of this QAPP). Room temperature, refrigerator, and freezer temperatures in long-term and short-term sample storage will be monitored.

3.7.3 Sample Tracking

Persons requiring samples from storage may initiate a sample transfer request. The sample custodian retrieves the samples requested and places them in the short-term, environmentally controlled storage unit or location indicated on the request. Following analysis or at the end of each day, the sample custodian will return the sample to the assigned environmentally controlled storage location.

3.7.4 Recordkeeping

Data related to sample preparation and analysis, as well as observations by laboratory analysts, will be recorded in bound laboratory notebooks or on designated laboratory sheets, as applicable. Raw data, hard copy or electronic, will undergo a secondary data review process. Hard copy raw data, including, but not limited to, the original chromatograms, worksheets, correspondence, and results shall be included with the data package submitted to the Project Manager.



**Table 3-1
Analytical Methodologies**

Analysis ⁽⁴⁾	Analytical Methods	Container	Preservation	Hold Time
SOIL				
COPCs	SW846 6010	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	6 months
TAL Metals	SW846 6010	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	6 months
Mercury	SW846 7471	10 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	None	28 days
TPH	EPA 1664 (SGT HEM)	100 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4°C	28 days
SVOCs	SW846 8270	30 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4°C	14 days
TAL PCBs	SW846 8082	30 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4°C	14 days
VOCs	SW846 8260	WideMouth Jar, TerraCore or EnCore ⁽¹⁾	4°C	14 days
TCLP Metals	SW846 1311, SW846 6010 &	100 grams, wide mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4°C	14 days/6 months ⁽²⁾
Cyanide	SW846 9012	4 oz soil jar	4°C	14 days
Chromium (VI)	SW846 7196	4 oz soil jar	None	30 days
Chromium (III)	SM 3500	4 oz soil jar	None	NA
Pesticides	SW846 8081	wide mouth Jar	4°C	14 days
Herbicides	SW846 8151	4 oz soil jar	4°C	14 days
Groundwater				
TAL Metals	SW846846 6010	250 mL, polyethylene or glass	HN0 ₃ to pH < 2	6 months
Mercury	SW846 7470	250 mL, polyethylene or glass	HN0 ₃ to pH < 2	28 days
TPH	EPA 1664 (SGT HEM)	1000 mL, glass with Teflon®-lined cap	4°C, H ₂ SO ₄ or HCl to pH < 2	28 days
SVOCs	SW846 8270	2-250 mL, glass with Teflon®-lined cap (amber)	4°C	7/40 days ⁽³⁾
TAL PCBs	SW846 8082	2-250 mL, glass with Teflon®-lined cap (amber)	4°C	7/40 days ⁽³⁾
VOCs	SW846 8260	3-40 mL, glass with Teflon®-lined septum	4°C, HCL	14 days

Notes:

COPCs - Constituents of Potential Concern

TAL - Target Analyte List

TPH - Total Petroleum Hydrocarbons

PCB - Polychlorinated biphenyls

VOCs - Volatile Organic Compounds

SVOCs - Semi-volatile Organic Compounds

SGT HEM - Silica Gel Treated N-Hexane Extractable Material

TCLP - Toxicity Characteristic Leaching Procedure

⁽¹⁾ There are a number of options for collecting soil samples for volatile analysis. The options include: EnCore® devices, TerraCore® devices, and wide mouth jars. Compliance with local regulatory requirements is necessary, and if dry weight determination is needed, a separate 2 oz jar must be collected.

⁽²⁾ 14 days for extraction, 180 days for analysis

⁽³⁾ 7 days for extraction, 40 days for analysis

⁽⁴⁾ Analysis of site samples to follow analyte list in Table 5-1 as pertaining to Chain of Custody instruction. Imported fill samples to follow analyte list in Table 5-2 as stated in DER-10, Technical Guidance for Investigation and Remediation (2010), Appendix 5 Subdivision 5.4(e): Allowable Constituent Levels for Imported Fill or Soil.



Table 3-2
Field Sample Identifiers

Sample Area	Sample Number	QC Type	Depth/Date code
Soil Sample Identifiers			
RES### = Residential Location	SS### = Soil Sample	0 - Primary Sample	### - Depth at Top of Sample
EA### = Residential Expansion Area	SB### = Soil Boring	1 - Duplicate Sample	(e.g. 2.0 feet is 020)
ROW = Right-of-Way Area	GS## = Garden Sample		
CPP = Corning-Painted Post School District Property	DP### = Disposal Profiling Sample		
CCA = Corning Christian Academy			
CMS = Memorial Stadium Area			
FCA = Flood Control Area			
Example ID: RES008SB005-0-002 (Primary soil sample collected from soil boring #5 at Residential Area Property 008 collected at 0.2 feet below ground surface.)			
Water Sample Identifiers			
CPP = Corning-Painted Post School District Property	MW## = Monitoring Well	0 - Primary Sample	YYMMDD - Date of Sampling
CMS = Memorial Stadium Area		1 - Duplicate Sample	(e.g. 1/1/2016 feet is 160101)
ROWEEC = Right-of-Way Area (Eastern End of Corning Blvd)			
CNY = Corning New York			
Example ID: CPPMW01-0-160101 (Primary groundwater sample collected from Monitoring Well #1 at the Corning-Painted Post School District Property on January 1, 2016.)			
QA/QC Field Sample Identifiers			
CNY = Corning New York ¹	TB## = Trip Blank	2 - Field Blank Sample	YYMMDD - Date of Sampling
	RB## = Rinse Blank	3 - Trip Blank	(e.g. 1/1/2016 feet is 160101)
Example ID: CNYRB01-2-160101 (First rinse blank collected on January 1, 2016.)			
Additional Field Sample Identifiers			
CNY = Corning New York ¹	IDW### = Investigative Derived Waste Sample	0 - Primary Sample	### - Depth at Top of Sample
SOD## = Sod Sample	C### = Composite Sample	1 - Duplicate Sample	(e.g. 2.0 feet is 020)
BP = Borrow Pit (Backfill Sample)	G### = Grab Sample		
Example ID: SOD01C001-0-000 (First composite soil sample [primary sample] collected at Sod Farm #1 collected at 0 feet below ground surface.)			

Notes:

¹CNY sample area identifier is only used for samples that are not specific to a specific sample area (i.e., rinse blank and/or IDW sample)

4. FIELD OPERATIONS

This section includes brief descriptions of field procedures used to conduct environmental evaluations in the project. Criteria or guidelines for choosing among alternatives are also included when more than one procedure can be used.

4.1 FIELD RECORDS

Documentation of field sampling will be performed to ensure data validity and facilitate analysis and evaluation. Examples of field documentation are provided in the following sections.

Field personnel are responsible for recording field activities in the appropriate field documentation logbooks or dedicated sampling sheets in sufficient detail to allow the significant aspects of the event to be reconstructed without relying on memory. It is the responsibility of the Field Team Manager to ensure that documents are complete and legible.

The field documentation forms or equivalent records that will be used during this investigation typically include the following:

- Soil sampling and borehole log forms;
- Field logbooks;
- Air monitoring records;
- Subsurface utility clearance records;
- Annotation of maps; and
- Sample chain-of-custody record.

4.1.1 Field Logbooks

Field logbooks will be used to record data collection activities. Activities will be described in as much detail as possible so that persons going to the facility can reconstruct a particular situation without relying on memory. Designated field sheets may also be used to record project activities.



A field logbook(s) will be maintained by the Field Team Manager or designated field team members present in the field to record information pertinent to daily activities, the field sampling program, and the equipment preparation efforts. Field logbooks will be bound, pages numbered, and entries made in permanent, waterproof ink. Field logbooks and field sheets will be scanned and transferred to the electronic project files or physically placed in the file at the end of field activities to provide a record of sampling.

Field logbooks and/or field sampling sheets will contain the following types of information, where applicable:

- Name and location of project.
- Date(s) and time(s) of sample collection.
- Name of Field Team Manager and/or other field team members.
- Field observations, including physical/environmental conditions during the field activity (i.e., weather).
- Summary of equipment preparation/decontamination procedures.
- Number, type, location, depth, and analysis required of samples taken and sample identification codes.
- A description of sampling methodologies or references to the PDI Work Plan and this QAPP.
- A cross-reference to photographs, if photographs are taken.
- Sample container/preservative.
- A cross-reference of sample identification codes or sampling points on annotated project maps or sketches.
- Sample shipping dates and methods.
- Deviations from the PDI Work Plan (if applicable).



Comments and other relevant observations such as weather conditions or other factors that may affect sample results or interpretation of sampling techniques and any modifications to sampling procedures as well as other technical comments regarding color, odor, texture, moisture and other sample characteristics.

4.1.2 Soil Sampling and Borehole Log Forms

Certain descriptive and sample information will be recorded during the completion of each boring and the collection/logging of soil samples. The information will be recorded in the field logbook, on a soil description form, borehole log form, or other appropriate form.

4.1.3 Corrections to Documentation

Field measurements made and samples collected will be recorded. Corrections will be made by drawing a line through the incorrect entry and writing in the correct entry. The person making the correction will date and initial the correction. There will be no erasures or deletions from the field logbooks.

4.2 SURVEYING

Any monitoring wells installed will be surveyed by a licensed surveyor, including horizontal coordinates, ground surface elevation, top of inner casing (riser) elevation, and top of outer protective casing elevation. The elevations will be reported to the nearest 0.01 foot.

Land-based survey methods will be used to establish a benchmark and a reference point to U.S. Geological Survey (USGS) datum. The data will be used along with depth to groundwater data to further define groundwater elevations within the Study Area.

Surface soil and soil boring locations will be recorded using a hand-held GPS unit with sub-meter accuracy.

4.3 ANNOTATION OF MAPS

Copies of Study Area base maps or sketches used by the field teams to record key Study Area conditions and to show approximate locations of soil borings, monitoring wells, buildings and



structures, utilities, and other appropriate project location information will be maintained (as needed) for the project files. The maps or sketches will be maintained by the Field Team Manager during field activities and transferred to the project files for a record of sampling locations.

4.4 AIR MONITORING

In accordance with the CAMP, air monitoring will be conducted to evaluate air quality during project activities (as needed). The data provided by the air monitoring could be used to determine the appropriate control actions and personal protective equipment (PPE) requirements.

Equipment calibration of air monitoring equipment will be performed in accordance with the manufacturer instructions.

A PID equipped with a 10.6 eV or an 11.7 eV lamp, calibrated with isobutylene, will be used to monitor the general area and the breathing zone of workers during intrusive activities to assess the potential presence of organic vapors.

4.5 FIELD CALIBRATION

Field instrumentation will be calibrated in accordance with the manufacturer supplied guidance manual to ensure that the instruments are operating properly and produce data that can satisfy the objectives of the sampling program. Specific field instruments that will be used during the project, when appropriate, include the following:

- Water level indicator;
- PID meter; and
- PM₁₀ Dust Monitor.

To ensure that the instruments are operating properly and are producing accurate and reliable data, routine calibration must be performed. Calibrations should be performed at a frequency recommended by the manufacturer. Calibration procedures are normally included with the equipment. Field calibrations should be performed at the beginning of the day and should be checked throughout the sampling day.



PID meters and PM₁₀ dust meters will be calibrated according to the instrument manufacturers' specifications. Daily calibrations will be performed by WESTON personnel. The recorded calibration information includes date of calibration, standards used, and calibration results will be recorded in the field logbook or designated field calibration sheets.

Groundwater Sampling Instruments

WESTON will use field instruments when conducting groundwater sampling activities. WESTON's field instrumentation will be calibrated to ensure that the instruments are operating properly and produce data that satisfy the objectives of the sampling program. Specific field instruments that will be used, when appropriate, include the following:

- pH meter;
- Conductivity meter;
- Turbidity meter;
- Thermometer or temperature sensor;
- Dissolved oxygen meter; and
- Oxidation-reduction potential meter.

If field calibration reveals that any of the instruments are outside established accuracy limits, the instrument will be serviced in the field according to the manufacturer's specifications as possible. If necessary, the instrument will be returned to the manufacturer for repair and servicing.



5. LABORATORY ANALYSIS

To generate analytical data of known and defensible quality, adherence to established QA protocols will be used. To ensure that the samples obtained in the field represent the particular environment from which they are collected and are of satisfactory quality, laboratory analysis will be performed in accordance with the PDI Work Plan SOPs as well as in accordance with TestAmerica laboratory SOPs established in TestAmerica's QAM provided in Attachment A of this QAPP.

5.1 LABORATORY REQUIREMENTS

TestAmerica (certified New York laboratory #10026 and certified New Jersey laboratory #11452) will perform analysis on environmental samples where certification exists. The laboratory will follow QA/QC procedures specified by the analytical methods.

5.2 METHOD DETECTION LIMITS

To generate data that meets the project-specific data quality objectives, the laboratory will demonstrate the sensitivities of the methodologies used for sample analyses will be at or below the method detection limits (i.e., MDLs). Tables 5-1 and 5-2 summarizes the laboratory MDLs.

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than non-detect (ND) and is determined from analysis of a sample in a given matrix containing the analyte. The MDL is estimated in an interference-free matrix, typically reagent water for water methods and a purified solid matrix (e.g., sand) for soil methods and shall be estimated for each compounds/analytes of interest using the procedures presented in 40 CFR, Part 136, Appendix B. The MDLs are extraction/digestion method-specific and includes any clean-up methods used. The laboratory performs MDL studies whenever the basic chemistry of the procedures changes. If any of the target analytes are not recovered, the MDL study will be repeated for the failed target analytes. The MDL study is performed at a minimum on an annual basis.



5.3 ANALYTICAL METHODS AND HOLDING TIMES

Table 3-1 summarizes the analytical methods to be used and the maximum holding times for water and soil samples. Sample holding times are calculated from the time of collection.

Samples collected under this QAPP will be analyzed using procedures of U.S. Environmental Protection Agency (USEPA) SW-846, 3rd Edition, Final Update V, 2015 (SW-846), USEPA Methods for Chemical Analyses of Waters and Wastes (MCAWW), or American Society of Testing and Materials (ASTM).

5.4 QUALITY CONTROL AND QUALITY ASSURANCE

Sample analyses will include a method blank, a method blank spike, a matrix spike, a laboratory duplicate for inorganic analyses (or matrix spike duplicate for organic analyses), and a laboratory control standard (inorganic analysis only) in each batch of 20 or fewer samples. In addition, appropriate surrogate compounds (organic analysis only) will be spiked into each sample. Recoveries from matrix spikes and surrogate compounds are calculated and recorded on control charts to maintain a history of system performance. The laboratory-performance-based acceptable limits for each compound/analyte will be established and provided by the laboratory.

Any blanks and/or other QC parameters not meeting the established acceptance criteria will prompt sample re-extraction/re-digestion and/or reanalysis as detailed in the laboratory SOPs that are included with TestAmerica's QA Manuals in Attachment A of this QAPP.

Before any instrument is used as a measurement device on the project samples, the instrument responses to known reference materials will be determined. The manner in which various instruments are calibrated is dependent upon the particular type of instrument and its intended use. Sample measurements are made within the calibrated range of the instrument. Preparation of reference materials used for calibration will be documented in the standards preparation notebook.

Instrument initial calibration will be performed to the pertinent method specifications or the manufacturing manual. Continuing calibration or calibration verification will be performed at frequencies outlined in the pertinent analytical methods. The acceptance criteria will be met before any samples are analyzed.



Details on TestAmerica's quality control/quality assurance program are provided in their QAMs (see Attachment A of this QAPP).

5.5 DATA REPORTING

Laboratory data deliverables will consist of analytical data in tabulated forms as well as the complete laboratory data deliverable package. TestAmerica will produce laboratory data packages which meet the requirements of NYSDEC Analytical Services Protocol (ASP) Category B (See DER-10 Appendix 2B Section 1.0b).

Additionally, TestAmerica will provide an EDD for all samples with QC sample data to be utilized during the data review/validation/report production activities.

5.6 DATA REVIEW/VALIDATION

All laboratory data deliverable packages will be reviewed for completeness, adherence to holding times, comparison with chain-of-custody, etc. Laboratory data package reviews may include the following activities:

- Review of laboratory supplied data package for completeness
- Review of chain-of-custody documents to verify sample identities.
- Review of sample log-in documents to identify any potential problems with custody seals, container integrity, sample preservation, labeling, etc.
- Review of sample analysis methods and holding times.
- Review of field blank and trip blank data to identify any potential problems with sampling devices contamination, sample container contamination, preservative contamination, laboratory reagent water contamination, or cross-contamination between samples during transport.
- Review of method blank data to determine the presence of any sources of contamination in the analytical process, where applicable.
- Review of MS/MSD data to evaluate the potential for matrix effects as a measure of analytical accuracy and sample homogeneity as a measure of analytical precision. MS/MSD



data will be compared to laboratory acceptance criteria for the maximum relative percent difference (RPD), where applicable.

- Review of laboratory control sample and laboratory control sample duplicate (LCS/LCSD) data as a measure of analytical accuracy and as a measure of analytical precision, where applicable. LCS/LCSD data will be compared to laboratory acceptance criteria for the maximum RPD.
- Review of laboratory control sample reference material (LCSRM) data as a measure of analytical accuracy, where applicable. LCSRM data will be compared to the certified acceptable ranges of analytical values.
- Review of sample and sample duplicate data as a measure of sample homogeneity and as a measure of analytical precision.
- Review of surrogate recovery data to assess analytical performance, where applicable. Surrogate recoveries will be compared to laboratory acceptance criteria to determine if they are within or outside of acceptable limits.
- Determine completeness as a percentage of measurements made which are judged to be valid measurements compared to the total number of measurements planned, where applicable.
- Review data summary sheets and qualifiers for consistency with raw data and qualifier definitions.

Data verification and/or validation will be performed by a qualified scientist, and a DAR or DUSR will be prepared in accordance with DER-10 Appendix 2B. The DAR or DUSR will provide the assessment included in the initial data review discussed above, with further related QA/QC information consideration, enabling evaluation of the analytical data's usability and quality as per DER-10 Appendix 2B requirements.

The data validation/review process will be documented through DARs/DUSRs and submission of the analytical data packages and DARs/DUSRs to the NYSDEC. Final and validated/reviewed analytical data, including applicable qualifiers will be summarized in tables for associated project summary reports.

Table 5-1
Study Area - Reporting Limits and Method Detection Limits

	Soil		Groundwater	
	RL	MDL	RL	MDL
COPCs [Method SW846 6010]	mg/Kg		mg/L	
Arsenic	2.00	0.400	0.0150	0.00555
Cadmium	0.200	0.0300	0.00200	0.000500
Lead	1.00	0.240	0.0100	0.00300
Metals [Method SW846 6010]	mg/Kg		mg/L	
Aluminum	10.0	4.40	0.200	0.0600
Antimony	15.0	0.400	0.0200	0.00679
Arsenic	2.00	0.400	0.0150	0.00555
Barium	0.500	0.110	0.00200	0.000700
Beryllium	0.200	0.0280	0.0200	0.00400
Boron	2.00	0.190	0.0200	0.00400
Cadmium	0.200	0.0300	0.00200	0.000500
Calcium	50.0	3.30	0.500	0.100
Chromium	0.500	0.200	0.00400	0.00100
Cobalt	0.500	0.0500	0.00400	0.000630
Copper	1.00	0.210	0.0100	0.00160
Iron	10.0	1.10	0.0500	0.0193
Lead	1.00	0.240	0.0100	0.00300
Magnesium	20.0	0.927	0.200	0.0434
Manganese	0.200	0.0320	0.00300	0.000400
Nickel	5.00	0.230	0.0100	0.00126
Potassium	30.0	20.0	0.500	0.100
Selenium	4.00	0.400	0.0250	0.00870
Silver	0.600	0.200	0.00600	0.00170
Sodium	140	13.0	1.00	0.324
Thallium	6.00	0.300	0.0200	0.0102
Vanadium	0.500	0.110	0.00500	0.00150
Zinc	2.00	0.153	0.0100	0.00150
Mercury [Method SW846 7471B/7470A]	mg/Kg		mg/L	
Mercury	0.0200	0.00810	0.000200	0.000120
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270]	µg/Kg		µg/L	
Biphenyl	170	28.2	5.00	0.653
bis (2-chloroisopropyl) ether	170	34.0	5.00	0.930
2,4,5-Trichlorophenol	170	46.0	5.00	0.490
2,4,6-Trichlorophenol	170	34.0	5.00	0.610
2,4-Dichlorophenol	170	18.0	5.00	0.630
2,4-Dimethylphenol	170	72.7	5.00	0.910
2,4-Dinitrophenol	1660	784	10.0	2.37
2,4-Dinitrotoluene	170	35.0	5.00	1.04
2,6-Dinitrotoluene	170	20.0	5.00	0.880
2-Chloronaphthalene	170	28.0	5.00	0.610
2-Chlorophenol	170	31.0	5.00	0.740
2-Methylphenol	170	20.0	5.00	0.880
2-Methylnaphthalene	170	34.0	5.00	1.29
2-Nitroaniline	330	25.0	10.0	0.650
2-Nitrophenol	170	48.0	5.00	0.590
3,3'-Dichlorobenzidine	330	200	5.00	1.04
3-Nitroaniline	330	47.0	10.0	0.820
4,6-Dinitro-2-methylphenol	330	170	10.0	2.20
4-Bromophenyl phenyl ether	170	24.0	5.00	1.02
4-Chloro-3-methylphenol	170	42.0	5.00	0.760
4-Chloroaniline	170	42.0	5.00	0.730
4-Chlorophenyl phenyl ether	170	21.0	5.00	0.960
4-Methylphenol	330	20.0	10.0	0.870
4-Nitroaniline	330	89.0	10.0	0.480
4-Nitrophenol	330	159	10.0	4.65
Acenaphthene	170	25.0	5.00	0.880
Acenaphthylene	170	22.0	5.00	0.650
Acetophenone	170	23.0	5.00	1.04
Anthracene	170	42.0	5.00	0.570
Atrazine	170	59.0	5.00	0.770
Benzaldehyde	170	135	5.00	0.860
Benzo[<i>a</i>]anthracene	170	27.6	5.00	0.550
Benzo[<i>a</i>]pyrene	170	25.0	5.00	0.470
Benzo[<i>b</i>]fluoranthene	170	27.0	5.00	0.440

Table 5-1
Study Area - Reporting Limits and Method Detection Limits
(continued)

	Soil		Groundwater	
	RL	MDL	RL	MDL
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270] (continued)	µg/Kg		µg/L	
Benzo[g,h,i]perylene	170	19.0	5.00	0.750
Benzo[k]fluoranthene	170	22.0	5.00	0.730
Bis(2-chloroethoxy)methane	170	36.0	5.00	0.690
Bis(2-chloroethyl)ether	170	22.0	5.00	0.400
Bis(2-ethylhexyl) phthalate	170	58.0	5.00	2.20
Butyl benzyl phthalate	170	28.0	5.00	1.00
Caprolactam	170	51.0	5.00	2.20
Carbazole	170	20.0	5.00	0.850
Chrysene	170	38.0	5.00	0.670
Dibenz(a,h)anthracene	170	30.0	5.00	0.420
Di-n-butyl phthalate	170	29.0	10.0	0.850
Di-n-octyl phthalate	170	20.0	5.00	1.00
Dibenzofuran	170	20.0	10.0	0.980
Diethyl phthalate	170	22.0	5.00	0.820
Dimethyl phthalate	170	20.0	5.00	0.690
Fluoranthene	170	18.0	5.00	0.720
Fluorene	170	20.0	5.00	0.800
Hexachlorobenzene	170	23.0	5.00	0.510
Hexachlorobutadiene	170	25.0	5.00	0.760
Hexachlorocyclopentadiene	170	23.0	5.00	0.610
Hexachloroethane	170	22.0	5.00	0.590
Indeno[1,2,3-cd]pyrene	170	22.0	5.00	0.470
Isophorone	170	36.0	5.00	0.670
N-Nitrosodi-n-propylamine	170	29.0	5.00	0.800
N-Nitrosodiphenylamine	170	138	5.00	0.510
Naphthalene	170	22.0	5.00	0.830
Nitrobenzene	170	30.0	5.00	0.740
Pentachlorophenol	330	170	10.0	2.20
Phenanthrene	170	25.0	5.00	0.650
Phenol	170	26.0	5.00	0.410
Pyrene	170	20.0	5.00	0.830
Volatile Organic Compounds (VOCs) [Method SW846 8260]	µg/Kg		µg/L	
1,1,1-Trichloroethane	5.00	0.363	1.00	0.820
1,1,2,2-Tetrachloroethane	5.00	0.811	1.00	0.210
1,1,2-Trichloro-1,2,2-trifluoroethane	5.00	1.14	1.00	0.310
1,1,2-Trichloroethane	5.00	0.650	1.00	0.230
1,1-Dichloroethane	5.00	0.610	1.00	0.380
1,1-Dichloroethene	5.00	0.612	1.00	0.290
1,2,4-Trichlorobenzene	5.00	0.304	1.00	0.410
1,2-Dibromo-3-Chloropropane	5.00	2.50	1.00	0.390
1,2-Dibromoethane	5.00	0.642	1.00	0.730
1,2-Dichlorobenzene	5.00	0.391	1.00	0.790
1,2-Dichloroethane	5.00	0.251	1.00	0.210
1,2-Dichloropropane	5.00	2.50	1.00	0.720
1,3-Dichlorobenzene	5.00	0.257	1.00	0.780
1,4-Dichlorobenzene	5.00	0.700	1.00	0.840
2-Butanone (MEK)	25.0	1.83	10.0	1.32
2-Hexanone	25.0	2.50	5.00	1.24
4-Methyl-2-pentanone (MIBK)	25.0	1.64	5.00	2.10
Acetone	25.0	4.21	10.0	3.00
Benzene	5.00	0.245	1.00	0.410
Bromodichloromethane	5.00	0.670	1.00	0.390
Bromotorm	5.00	2.50	1.00	0.260
Bromomethane	5.00	0.450	1.00	0.690
Carbon disulfide	5.00	2.50	1.00	0.190
Carbon tetrachloride	5.00	0.484	1.00	0.270
Chlorobenzene	5.00	0.660	1.00	0.750
Chloroethane	5.00	1.13	1.00	0.320
Chlorotorm	5.00	0.309	1.00	0.340
Chloromethane	5.00	0.302	1.00	0.350
cis-1,2-Dichloroethene	5.00	0.640	1.00	0.810
cis-1,3-Dichloropropene	5.00	0.720	1.00	0.360
Cyclohexane	5.00	0.700	1.00	0.180
Dibromochloromethane	5.00	0.640	1.00	0.320
Dichlorodifluoromethane	5.00	0.413	1.00	0.680



Table 5-1
Study Area - Reporting Limits and Method Detection Limits
(continued)

	Soil		Groundwater	
	RL	MDL	RL	MDL
<i>Volatile Organic Compounds (VOCs) [Method SW846 8260](continued)</i>	ug/Kg		ug/L	
Ethylbenzene	5.00	0.345	1.00	0.740
Isopropylbenzene	5.00	0.754	1.00	0.790
Methyl acetate	5.00	0.930	2.50	1.30
Methyl tert-butyl ether	5.00	0.491	1.00	0.160
Methylcyclohexane	5.00	0.760	1.00	0.160
Methylene Chloride	5.00	2.30	1.00	0.440
Styrene	5.00	0.250	1.00	0.730
Tetrachloroethene	5.00	0.671	1.00	0.360
Toluene	5.00	0.378	1.00	0.510
trans-1,2-Dichloroethene	5.00	0.516	1.00	0.900
trans-1,3-Dichloropropene	5.00	2.20	1.00	0.370
Trichloroethene	5.00	1.10	1.00	0.460
Trichlorofluoromethane	5.00	0.473	1.00	0.880
Vinyl chloride	5.00	0.610	1.00	0.900
Xylenes, Total	10.0	0.840	2.00	0.660
<i>TCLP Metals [Method SW846 6010 / 7470]</i>	mg/L		--	
Arsenic	0.0150	0.00555	--	--
Barium	1.00	0.100	--	--
Cadmium	0.00200	0.000500	--	--
Chromium	0.0200	0.0100	--	--
Lead	0.0200	0.00300	--	--
Mercury	0.000200	0.000120	--	--
Selenium	0.0250	0.00870	--	--
Silver	0.00600	0.00170	--	--

Notes:

mg/Kg - milligram per kilogram

µg/Kg - microgram per kilogram

mg/L - milligram per liter

µg/L - microgram per liter

RL - Reporting Limit

MDL - Method Detection Limit

Listed limits are the highest current MDL and RL inclusive of TestAmerica Buffalo and TestAmerica Edison laboratories for standard analytical testing.

**Table 5-2
Imported Fill - Reporting Limits and Method Detection Limits**

	Soil		Water	
	RL	MDL	RL	MDL
Metals [Method SW846 6010]	mg/Kg		--	
Arsenic	3.00	0.983	--	--
Barium	40.0	1.43	--	--
Beryllium	0.400	0.339	--	--
Cadmium	0.800	0.417	--	--
Copper	5.00	1.30	--	--
Lead	2.00	0.785	--	--
Manganese	3.00	1.05	--	--
Nickel	8.00	1.46	--	--
Selenium	4.00	1.38	--	--
Silver	2.00	0.353	--	--
Zinc	6.00	1.46	--	--
Mercury [Method SW846 7471]	mg/Kg		--	
Mercury	0.020	0.012	--	--
Cyanide [Method SW846 9012]	mg/Kg		--	
Cyanide	1.00	0.483	--	--
Chromium [Method SW846 7196]	mg/Kg		--	
Chromium, Hexavalent	2.00	0.395	--	--
Chromium, Trivalent	2.00	0.630	--	--
Pesticides [Method SW846 8081]	µg/Kg		--	
4,4'-DDD	6.70	0.880	--	--
4,4'-DDE	6.70	0.970	--	--
4,4'-DDT	6.70	0.690	--	--
Aldrin	6.70	0.810	--	--
Alpha-BHC	2.00	0.610	--	--
Chlordane (alpha)	6.70	1.09	--	--
Beta-BHC	2.00	0.650	--	--
Delta-BHC	2.00	0.730	--	--
Dieldrin	2.00	0.870	--	--
Endosulfan I	6.70	0.930	--	--
Endosulfan II	6.70	1.05	--	--
Endosulfan sulfate	6.70	0.780	--	--
Endrin	6.70	0.850	--	--
Lindane	2.00	0.600	--	--
Heptachlor	6.70	0.860	--	--
Polychlorinated Biphenyls (PCBs) [Method SW846 8082]	µg/Kg		--	
PCB-1016	67.0	8.90	--	--
PCB-1221	67.0	8.90	--	--
PCB-1232	67.0	8.90	--	--
PCB-1242	67.0	8.90	--	--
PCB-1248	67.0	8.90	--	--
PCB-1254	67.0	9.20	--	--
PCB-1260	67.0	9.20	--	--
Polychlorinated biphenyls	67.0	9.20	--	--
Herbicides [Method SW846 8151]	µg/Kg		--	
2,4,5-TP Acid (Silvex)	17.0	11.4	--	--
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270]	µg/Kg		--	
Acenaphthene	330	25.0	--	--
Acenaphthylene	330	22.0	--	--
Anthracene	330	42.0	--	--
Benzo(a)anthracene	170	27.6	--	--
Benzo(a)pyrene	170	25.0	--	--
Benzo(b)fluoranthene	170	27.0	--	--
Benzo(g,h,i)perylene	330	19.0	--	--
Benzo(k)fluoranthene	170	22.0	--	--
Chrysene	330	38.0	--	--
Dibenz(a,h)anthracene	170	30.0	--	--
Dibenzofuran	330	20.0	--	--
Fluoranthene	330	18.0	--	--
Fluorene	330	20.0	--	--
Hexachlorobenzene	170	23.0	--	--
Indeno(1,2,3-cd)pyrene	170	22.0	--	--
m-Cresol(s)	330	20.0	--	--
Naphthalene	330	26.0	--	--
o-Cresol(s)	330	20.0	--	--
p-Cresol(s)	330	22.0	--	--
Pentachlorophenol	330	170	--	--
Phenanthrene	330	25.0	--	--



Table 5-2
Imported Fill - Reporting Limits and Method Detection Limits
(continued)

	Soil		Water	
	RL	MDL	RL	MDL
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270] (continued)	µg/Kg		--	
Phenol	330	26.0	--	--
Pyrene	330	20.0	--	--
1,4-Dioxane	330	88.4	--	--
Volatile Organic Compounds (VOCs) [Method SW846 8260]	µg/Kg		µg/L	
1,1,1-Trichloroethane	100	0.380	1.00	0.820
1,1-Dichloroethane	100	0.610	1.00	0.380
1,1-Dichloroethene	100	0.612	1.00	0.340
1,2-Dichlorobenzene	100	0.391	1.00	0.790
1,2-Dichloroethane	100	0.251	1.00	0.250
1,2-Dichloroethene(trans)	100	0.640	1.00	0.810
1,2-Dichloroethene(cis)	100	0.516	1.00	0.900
1,3-Dichlorobenzene	100	0.257	1.00	0.780
1,4-Dichlorobenzene	100	0.700	1.00	0.840
1,4-Dioxane	1900	21.8	50.0	9.32
Acetone	500	4.210	10.0	3.00
Benzene	100	0.245	1.00	0.410
Butylbenzene	100	0.435	1.00	0.640
Carbon tetrachloride	100	0.484	1.00	0.330
Chlorobenzene	100	0.660	1.00	0.750
Chloroform	100	0.309	1.00	0.340
Ethylbenzene	100	0.345	1.00	0.740
Methyl ethyl ketone	500	1.83	10.0	2.20
Methyl tert-butyl ether	100	0.491	1.00	0.160
Methylene chloride	100	2.30	1.00	0.440
m-Cresol(s)	200	0.840	2.00	0.660
o-Cresol(s)	100	0.653	1.00	0.760
Propylbenzene-n	100	0.400	1.00	0.690
Sec-Butylbenzene	100	0.435	1.00	0.750
Tert-Butylbenzene	100	0.520	1.00	0.810
Tetrachloroethene	100	0.671	1.00	0.360
Toluene	100	0.378	1.00	0.510
Trichloroethene	100	1.10	1.00	0.460
Trimethylbenzene-1,2,4	100	0.960	1.00	0.750
Trimethylbenzene-1,3,5	100	0.322	1.00	0.770
Vinyl chloride	100	0.610	1.00	0.900
Xylene (mixed)	200	0.840	2.00	0.660

Notes:

mg/Kg - milligram per kilogram

µg/Kg - microgram per kilogram

mg/L - milligram per liter

µg/L - microgram per liter

RL - Reporting Limit

MDL - Method Detection Limit

Listed limits are the highest current MDL and RL inclusive of TestAmerica Buffalo and TestAmerica Edison laboratories for standard analytical testing.



6. REFERENCES

U.S. Environmental Protection Agency (USEPA) *Guidance for Quality Assurance Project Plans* (EPA-QA/G-5, 2002), December 2002.

EPA Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA-QA/G-4), Feb 2006.

EPA Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA Publication SW-846. Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015).



ATTACHMENT A

TESTAMERICA'S QUALITY ASSURANCE MANUAL AND STANDARD OPERATING PROCEDURES

Cover Page:

Quality Assurance Manual

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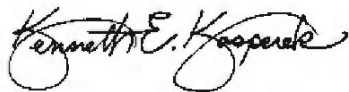
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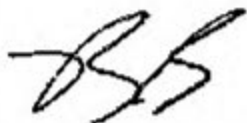
**Title Page:
Quality Assurance Manual
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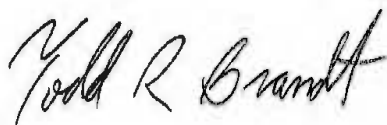
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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-002	Subcontracting Procedures
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CA-Q-S-004	Method Compliance & Data Authenticity Audits

REFERENCED LABORATORY SOPs

SOP Reference	Title
BF-GP-001	Calibration of Autopipettes and Repipetters
BF-GP-002	Support Equipment: Maintenance, Record Keeping and Corrective Actions
BF-GP-005	Sample Homogenization and Subsampling
BF-GP-012	Technical Data Review
BF-GP-013	Manual Integration
BF-GP-015	Record Storage and Retention
BF-GP-018	Strict Internal Chain of Custody

BF-GP-019	Standard Traceability and Preparation
BF-GP-020	Thermometer Calibration
BF-PM-001	Project Information Requirements
BF-PM-003	Bottle Order Set-up
BF-PM-005	Correctness of Analysis
BF-PM-008	Massachusetts DEP Notification Procedures
BF-QA-001	Determination of Method Detection Limits
BF-QA-002	Quality Control Limits
BF-QA-003	Procedure for Writing, Reviewing and Revising Controlled Documents
BF-QA-004	Laboratory Personnel Training
BF-QA-005	Preventative and Corrective Action
BF-QA-006	Data Quality Review
BF-SR-001	Cooler Shipping - Bottle Kits and Samples
BF-SR-002	Receipt of Analytical Samples

- The full list of Laboratory SOPs is maintained in the Quality Assurance Department
- The full list of analytical methods performed in the Laboratory is can be exported from the Laboratory Information Management System's Total Access Database

SECTION 3

INTRODUCTION, SCOPE AND APPLICABILITY

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Buffalo's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards, The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water*, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986, Final Update I, July 1992, Final Update II A, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261. New York State Analytical Services Protocol, July 2005
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005).
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 19th, 20th, and on-line Editions. 21st.
- U.S. Department of Energy Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- U.S. Department of Energy Order 414.1C, *Quality Assurance*, June 17, 2005.
- U.S. Department of Energy, *Quality Systems for Analytical Services*, Revision 3.6, November 2010.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Section 19.0. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director/Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. The manual itself is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing

regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & updating procedures (refer to BF-QA-003)

SECTION 4

MANAGEMENT REQUIREMENTS

4.1 OVERVIEW

TestAmerica Buffalo is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer, Executive VP Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Buffalo is presented in Figure 4-1.

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Buffalo laboratory.

4.2.2 Laboratory Director

TestAmerica Buffalo's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

Specific responsibilities include, but are not limited to:

- Provides one or more department managers for the appropriate fields of testing. If the Department Manager is absent for a period of time exceeding 15 consecutive calendar

days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Department Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary NELAC accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Leads the management team, consisting of the QA Manager, the Technical Manager, and the Operations Manager as direct reports.

4.2.2 Quality Assurance (QA) Manager or Designee

The QA manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA department to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems, data authenticity and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a subset of all final data reports for internal consistency.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Leads the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the

QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.

- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025.

4.2.3 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.

4.2.4 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.5 Department Managers

Department Managers report to the Operations Manager. The Department Managers serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, and development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Human Resources

Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.6 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

4.2.7 Environmental Health & Safety Coordinator

The Environmental Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Safety Data Sheet (SDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.8 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.

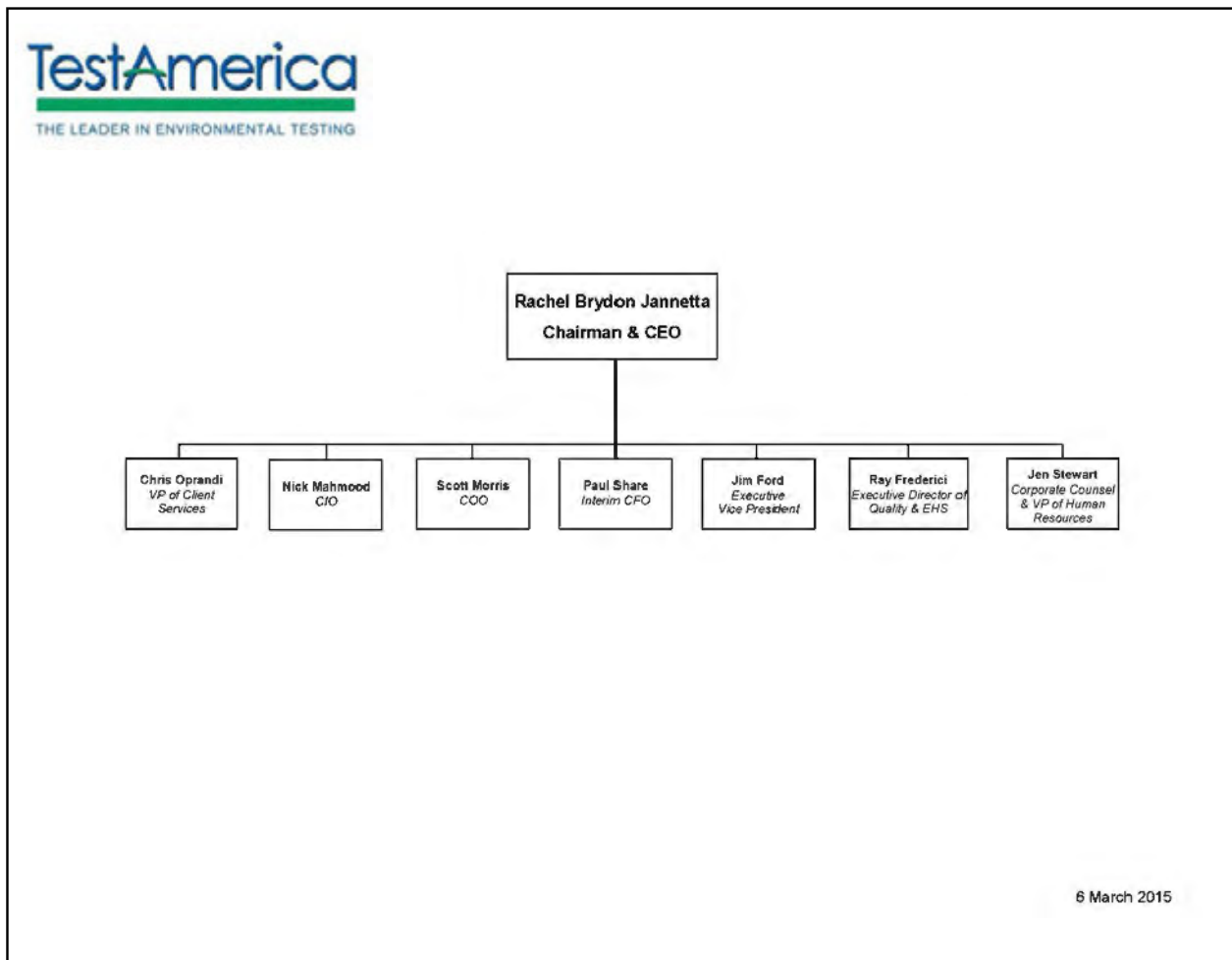
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

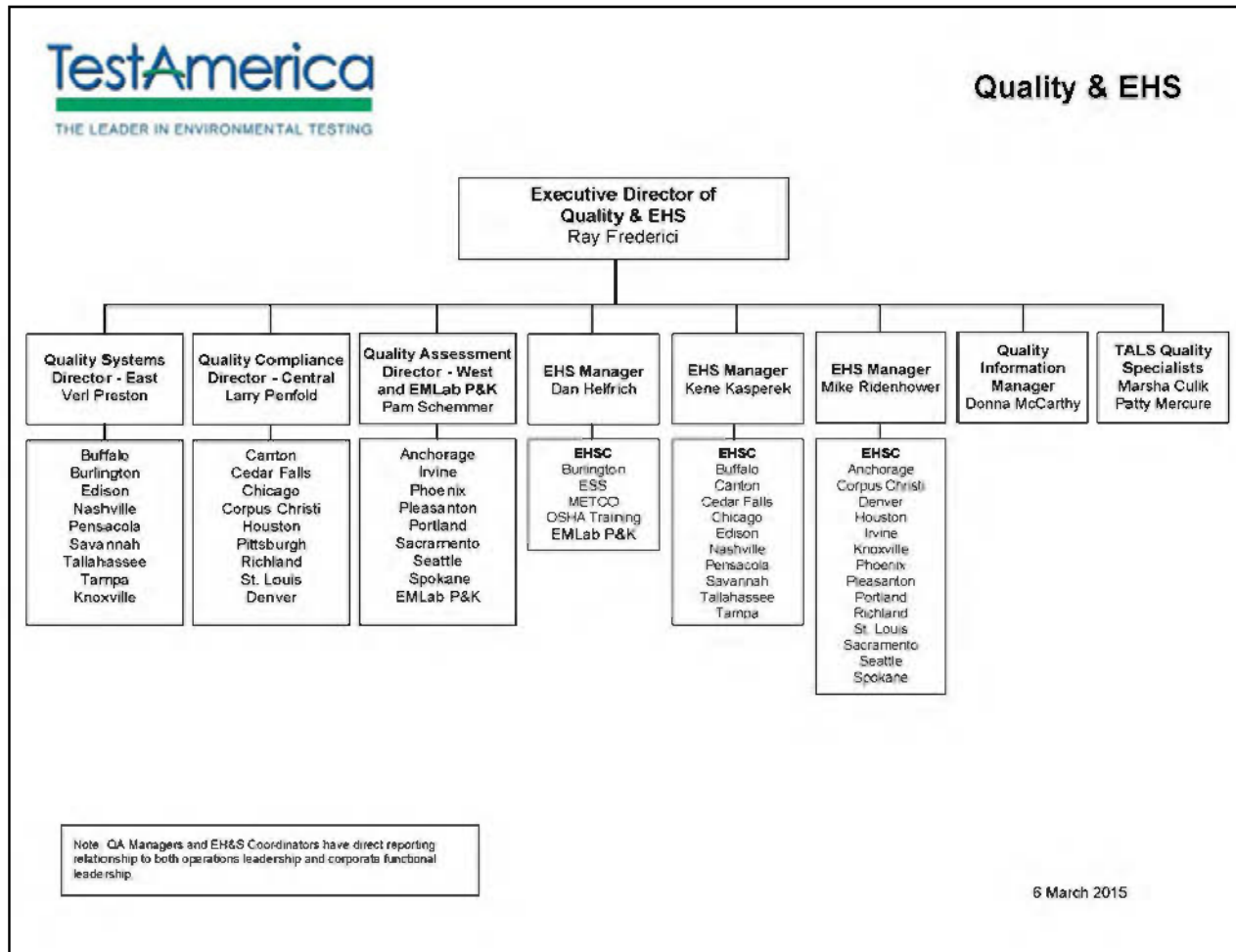
4.3 **DEPUTIES**

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director	Operations Manager (1) Technical Manager (2)	
QA Manager	QA Specialist (1) Operations Manager (2)	
Technical Manager	Laboratory Director (1) Operations Manager (2)	
Operations Manager	Department Manager (1) Department Manager (2)	Selected based on availability
Manager of Project Management	Project Manager (1) Client Services Director (2)	Selected based on availability
Project Manager	Project Manager (1) Project Management Asst. (2)	(1) 2 ^o team PM (2) Team PMA
Organic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Inorganic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Data Validation / Data Packaging Manager	Data Validation Specialist Data Packaging Specialist	Selected based on department and availability
EHS Coordinator	Laboratory Director (1) EHS Manager (2)	
Sample Management Manager	Sample Custodian (1) EHS Coordinator (2)	
Bottle Preparation / Shipping Manager	Bottle Prep Technician (1) Sample Mng't Manager (2)	

Figure 4-1.
Corporate and Laboratory Organization Charts







SECTION 5

QUALITY SYSTEM

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- To comply with the NELAC Standards (2003), ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).

- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents:

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratories normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "*analytical quality control*". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains Quality Control Limit Data in their LIMS system. A summary report is generated from LIMS to check the precision and accuracy acceptability limits for performed analyses on request. The summary report is generated and is managed by the laboratory's QA department. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 24.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The procedure for determining the statistical limits may be found in SOP BF-QA-002, Quality Control Limits. The analysts are instructed to use the current limits in the laboratory (dated and approved the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory through date sensitive tables within the LIMS System. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

The QA Manager periodically evaluates these to determine if adjustments need to be made or for corrective actions to methods (SOP No. BF-QA-002). All findings are documented and kept on file.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. BF-QA-003.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action notices. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item, or an 'end of document' page, the effective date, revision number and the laboratory's name. The Quality personnel are responsible for the maintenance of the system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a Department Manager submits an electronic draft to the QA Department for

suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years for the majority of procedures. Exceptions include review every 1 year for Drinking Water programs and the Kentucky CWA program. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory Intranet site and is available to all personnel.

For changes to SOPs, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents".

Forms, worksheets, work instructions and information are organized by department and are maintained electronically by QA. There is a table of contents. As revisions are required, a new version number and revision date is assigned. Controlled electronic copies are made available on a public server for laboratory staff to access.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. BF-GP-015.

SECTION 7

SERVICE TO THE CLIENT

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relations Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contact Administrator
- VP of Operations
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts. The Project Managers at the TestAmerica Buffalo facility also maintains copies of these documents.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal a PM is assigned to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers/Designees are available to discuss any technical questions or concerns that the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOP’s on Subcontracting Procedures (CA-L-S-002).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, may require notification prior to placing such work.

Approval may be documented through reference in a quote / contract or e-mail correspondence.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Account Executive (AE) or Client Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable (e.g. on the subcontractors TNI, A2LA accreditation or State certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work-sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager/Designee begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and
- Corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors/Managers, QA Managers and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM (or AE or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontract Laboratory Certification Verification Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared

within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data are incorporated into the laboratories EDD (i.e. imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. . The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1 Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: _____
Laboratory: _____
Address: _____
Contact and e-mail address: _____
Phone: Direct _____ Fax _____

Requested Item ³	Date Received	Reviewed/ Accepted	Date
1. Copy of State Certification ¹			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program ³			
5. QA Manual ³			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response ^{1,3}			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit) ³			
8. Sample Report ³			
9. SOQ or Summary list of Technical Staff and Qualifications ³			
10. SOPs for Methods to Be Loadshifted ^{2,3}			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			

1 - Required when emergency procedures are implemented.

2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.

3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: _____ By Whom: _____

Comments: _____

Lab Acceptable for Subcontracting Work: YES NO Limitations: _____

QA Manager (Signature): _____ Date: _____

☐ Forwarded to Contract Coordinator, by: _____ Date: _____

SECTION 9

PURCHASING SERVICES AND SUPPLIES

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001 and TestAmerica Buffalo SOP on Solvent Purity, SOP BF-OP-013.

9.3.1 Purchasing

Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Purchase requisitions are

placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

9.3.2 Receiving

It is the responsibility of the purchasing manager/designee to receive the shipment. It is the responsibility of the department that ordered the materials to date the material when received. Once the ordered reagents or materials are received, the department that submitted the order compares the information on the label or packaging to the original order to ensure that the purchase meets quality level specified. Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot not be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- umho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in the LIMS system, files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. DOC No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers, Operations Manager and/or Technical Manager.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurements & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the

problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (available on the intranet site).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Manager are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS

10.1 OVERVIEW

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, e.g., communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in the laboratory SOP related Corrective Action (BF-QA-005).

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to BF-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for resolution. The department manager may elect to discuss it with the Technical Manager, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's non-conformance and corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director, Technical Manager, Operations Manager or QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with the analytical method requirements and the reason.

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Manager, the Operations Manager or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due

to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's non-conformance and corrective action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Manager, and QA Manager. Suspected misrepresentation issues may also be reported to any member of the corporate staff as identified in Ethics Policy, CA-L-P-001. The data integrity hotline (1-800-736-9407) may also be used. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & EHS and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12

CORRECTIVE ACTION

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memo (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Project Management concerns regarding specific analytical results
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and External Audit Findings

- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. A NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Operations Manager, Technical Manager, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into the Laboratory Information Management System (LIMS) and each CAR is entered into a spreadsheet (Figure 12-1) for tracking and trending purposes for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.
- Also refer to Section 15.1.4, Special Audits)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of a NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, work instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly at a minimum by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example – Corrective Action Notice

TestAmerica Buffalo																			
Corrective Action Summary																			
#	Source	Type	Audit Organization	Dept.	Method	Repeat Finding?	Category	Finding, Deficiency, Area Needing Improvement or Recommended Action	Laboratory Investigation Summary	Root Cause of Deficiency	Laboratory Corrective Action Plan	Resp. Person	Date Opened	Response Due	CA Due Date	Date Lab Closed	Follow up notes	Follow Up Date	Follow-up Closed By
1																			
2																			
3																			
4																			
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Table 12-1.

Example – General Corrective Action Procedures

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank (Analyst)	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards (Analyst, Department Manager)	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) (Analyst, Department Manager)	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards (Analyst, Data Reviewer)	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) (Analyst, Data Reviewer)	- % Recovery within limits documented in LIMs.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for the data for that sample shall be reported with qualifiers.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) (Analyst, Data Reviewer)	- % Recovery within limits specified in LIMS.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) When the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates (Analyst, Data Reviewer)	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) (Analyst, Data Reviewer)	< Reporting Limit [±]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples (QA Manager, Department Manager)	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Internal / External Audits (QA Manager, Department Manager, Operations Manager, Technical Manager, Laboratory Director)	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints (Project Managers, Lab Director, Sales and Marketing, QA Manager)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director, Operations Manager, Department Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (EH&S Coordinator, Lab Director, Operations Manager, Department Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through EH&S office.

Note: 1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the

ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and the other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13.0

PREVENTIVE ACTION / IMPROVEMENT

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management system reviews, review of monthly QA Metrics Report, evaluation of internal or external audits, results & evaluations of proficiency testing (PT) performance, review of control charts and QC results, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's Corrective Action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.

- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review

13.1.2 Any Preventive Actions/Process Improvements undertaken or attempted shall be taken into account during the Annual Management Systems Review (Section 17). A highly detailed report is not required; however a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

SECTION 14.0

CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention specify additional storage, archiving and retention procedures.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Hardcopy technical records are maintained by the Laboratory Director and the QA Department while electronic technical records are maintained by the IT Administrator.

Table 14-1. Record Index¹

	<u>Record Types¹:</u>	<u>Retention Time:</u>
Technical Records	- Raw Data - Logbooks² - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - Policy Memorandums - SOPs - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* Data Investigation: 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types¹:	Retention Time:
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP -SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	All HR docs have different retention times: Refer to HR Manual
	Administrative Policies Technical Training Records	7 years

¹ Record Types encompass hardcopy and electronic records.

² Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained according to BF-GP-015 and in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement. All records shall be protected against fire, theft, loss, environmental deterioration and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees and shall be documented with an access log.

If records are archived off-site they are to be stored in a secure location where a record is maintained of any entry into the storage facility.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Specific Information related to archival of data for greater than 5 years may be found in TestAmerica Buffalo SOP BF-GP-015.

Table 14-2. Special Record Retention Requirements

Program	¹ Retention Requirement
Drinking Water – All States	5 years (project records) 10 years-Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	5 years
NY Potable Water NYCRR Part 55-2	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

¹Note: Extended retention requirements are noted with the archive documents or addressed in TestAmerica Buffalo facility-specific records retention procedure BF-GP-015.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (any records stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory

transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the Job Number in TALS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to

enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

14.2.2 Observations, data and calculations are recorded real-time.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries.
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

14.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

- 14.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.
- 14.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.
- 14.5.4** The laboratory has a record management system (also known as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in LIMS and logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.
- 14.5.5** Records are considered archived when noted as such in the records management system (also known as document control). Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned.

14.5.6 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.7 Records Disposal

- 14.5.7.1** Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).
- 14.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS

15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-003)	Methods Audits Frequency: All methods are reviewed annually 50% of methods receive a QA Technical Audit 50% of methods receive a SOP Method Compliance Audit
Special	QA Department or Designee	Surveillance or spot checks performed as needed to monitor specific issues
Performance Testing	Coordinated by Corporate QA	Two successful per year for each TNI -NELAC field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The

audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, Chrom AuditMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil, and Air.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. . When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Director prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Manager, Operations Manager, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-004 & Work Instruction No. CA-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.

- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
 - Adequacy of staff, equipment and facility resources.
 - Adequacy of policies and procedures.
 - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes.

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The TestAmerica Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, VP of Quality, Technical & Operations Support, VP of Client and Technical Services, VPs of Operations and Quality Directors receive a monthly report from the Exec. Director of Quality & EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located in the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers/Department Managers – <u>General</u>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- The Human Resource office maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in TestAmerica Buffalo SOP BF-QA-004, Laboratory Personnel Training.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive

training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy No. CW-L-P-004 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 OVERVIEW

TestAmerica Buffalo is a 32,000 ft² secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for field operations, bottle kit preparation, sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Key equipment has been provided with back-up power supply in the event of a power outage.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building pass cards and alarm codes are distributed to all facility employees.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. [The reason for this is that it is important to know who is in the building in case of a safety emergency. The visitors logbook is used to ensure that everyone got out of the building safely.] In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and

vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19.0

TEST METHODS AND METHOD VALIDATION

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory:

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and Laboratory SOP BF-QA-003, Procedure for Writing, Reviewing and Revising Controlled Quality Documents (QAM, SOP, etc)
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

19.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; May 18, 2012
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994
- NIOSH Manual of Analytical Methods, 4th ed., August 1994.
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th/21st/22nd/on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261
- New York State DEC Analytical Services Protocol, 2005
- New York State DOH Methods Manual
- Massachusetts Contingency Plan 310 CMR 40, April 25, 2014
- Connecticut Reasonable Confidence Protocol, July 2006

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability (BF-QA-004) is performed whenever there is a significant change in instrument type (e.g., new instrumentation), method or personnel.

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager/Designee and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

- 19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.
- 19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.
- 19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- 19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.
- 19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.
- 19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- 19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:
- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
 - Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision

guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each

of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. BF-QA-001 for details on the laboratory's MDL process.

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

19.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirement.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time

window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory's SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, and specific electrode response factors.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty”: the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor $k=2$.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of $k = 3$. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ± 0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as "reanalysis") may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples $\leq 5x$ the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor or Laboratory Director/Manager if unsure.

19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the 'TALS Data System' which is a LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes a SQL server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, and data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls such as password protection or website access approval, when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review sheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- 19.14.2.1** All raw data must be retained in the project job folder, computer file, and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.
- 19.14.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ($\mu\text{g/l}$) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ($\mu\text{g/kg}$) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.
- 19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, final inorganic results are reported to 2 significant figures for values less than 10 and 3 significant figures for values greater than 10 on the final report. Organic results are generally reported to 1 significant figure for values less than 10 and 2 significant figures for values greater than 10 on the final report. The number of significant figures may be adjusted based on client or project requirements.
- 19.14.2.4** For those methods that do not have an instrument printout, an instrumental output or a calculation spreadsheet upload compatible with the LIMS System, the final results and dilution factors are entered directly into LIMS by the analyst, and the software formats the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- 19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is automatically transferred to the network server and, eventually, to a back-up tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"ed out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several laboratory SOPs (e.g. BF-SR-002, "Receipt of Analytical Samples", BF-GP-012, "Technical Data Review", and BF-PM-001, "Project Information Requirements") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (BF-GP-013, Manual Integration). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review –The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. When complete, the report is issued to the client.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder

needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

- 19.14.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.
- 19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1.
Example - Demonstration of Capability Documentation



TESTAMERICA LABORATORIES, INC.

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

BF-QA-DOC-004
DOC Cert. Statement
Rev.0 2/27/13

Employee: _____

Page _____ of _____

Method Number: _____

Date: _____

Parameters or Analytes: _____

Initial Demonstration of Capability: ☐

SOP Number: _____

Revision # _____

Date Read _____

Trained By: _____

Date training began: _____

Date training completed: _____

Continued Demonstration of Capability: ☐

SOP Number: _____

Revision # _____

Date Read _____

I CERTIFY that I have read, understand and agree to use the SOP identified above. I have also submitted data associated with the demonstration of capability.

Employee Signature

Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

Operations Manager

Signature

Date

Quality Assurance Manager

Signature

Date

SECTION 20

EQUIPMENT (AND CALIBRATIONS)

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrumentation records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses

20.2.6 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance. Laboratory SOPs BF-GP-001, "Calibration of Autopipettes and Repipettors" and BF-GP-002, "Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devices and Reagent Water" provide additional detail on the monitoring and record keeping for support equipment.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All reusable thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use. Disposable thermometers are discarded upon expiration and replaced with newly purchased thermometers. IR thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual. The IR thermometers are verified daily and calibrated quarterly. Digital probes and thermocouples are calibrated quarterly.

The NIST Mercury thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST digital thermometer is recalibrated every one year (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories) and have ranges applicable to method and certification

requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP BF-GP-020, "Thermometer Calibration".

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between $> 0^{\circ}\text{C}$ and $\leq 6^{\circ}\text{C}$.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically at a minimum on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly (or if not utilized monthly, immediately prior to its usage) by setting the sample volume to 100ml and recording the volume received. The results are filed

in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

Additional calibration and use information is detailed in laboratory SOP BF-FS-006, "Calibration of Field Meter".

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

20.4.1.1 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

- 20.4.1.2** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- 20.4.1.3** The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules are methods where the referenced method does not specify two or more standards.
- 20.4.1.4** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met i.e., RPD, per NELAC (2003) Standard, Section 5.5.5.10 and 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a).when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b).when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.2.1 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See laboratory SOP's BF-MB-005 and BF-MV-007 for guidelines for making tentative identifications

Note:

For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation – TestAmerica Buffalo

 TestAmerica Buffalo Instrument List					
Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS Instrumentation	Agilent	5975	US83110163	2013	good
GC/MS Instrumentation	Agilent	5973	US02450141	2013	good
GC/MS Instrumentation	Agilent	5975	CN10833020	2009	good
GC/MS Instrumentation	Agilent	5975	US80838844	2008	good
GC/MS Instrumentation	Agilent	5973	US44621446	2005	good
GC/MS Instrumentation	Agilent	5973	US52420646	2005	good
GC/MS Instrumentation	Agilent	5973	US41720721	2004	good
GC/MS Instrumentation	Agilent	5973	US35120354	2004	good
GC/MS Instrumentation	Agilent	5973	US41720707	2004	good
GC/MS Instrumentation	Agilent	5973	US10241053	2003	good
GC/MS Instrumentation	Agilent	5973	US30965634	2003	good
GC/MS Instrumentation	Agilent	5973	US03965692	2003	good
GC/MS Instrumentation	Agilent	5973	US05060076	2001	good
GC/MS Instrumentation	Agilent	5973	US05060084	2001	good
GC/MS Instrumentation	Agilent	5973	US03950346	2001	good
GC/MS Instrumentation	Agilent	5973	US82321636	2001	good
GC Instrumentation	Perkin Elmer	Claruss 608 dual uECD	680S10042901	2012	good
GC Instrumentation	Perkin Elmer	Claruss 600 dual FID	665S10020401	2012	good
GC Instrumentation	Agilent	6890 dual uECD	CN10520009	2005	good
GC Instrumentation	Agilent	6890 dual uECD	CN10520010	2005	good
GC Instrumentation	Agilent	6890 dual uECD	CN10448015	2005	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A53126	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A63465	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A53464	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A53463	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A54409	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A54408	1994	good
GC Instrumentation	Hewlett Packard	5890II FID/FID	3115A34892	1994	good
GC Instrumentation	Hewlett Packard	5890II PID/FID	3336A60622	1994	good
GC Instrumentation	Hewlett Packard	5890II Hal/PID	3235A54089	1994	good
GC Instrumentation	Hewlett Packard	5890II PID/FID	3336A53465	1994	good
GC Instrumentation	Hewlett Packard	5890II dual FID	3336A53727	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3310A47661	1993	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A53325	1993	good

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GC Instrumentation	Hewlett Packard	5890II PID/FID	3133A37157	1993	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3203A42206	1992	good
GC Instrumentation	Hewlett Packard	5890II dual FID	3019A28433	1991	good
GC Instrumentation	Hewlett Packard	5890II Hall/PID	3121A35782	1990	good
Metals Instrumentation	Perkin Elmer	Elan 9000 ICP-MS	P0230202	2002	good
Metals Instrumentation	Leeman	PS200 II	HG9045	2000	good
Metals Instrumentation	Leeman	PS200 II	HG0033	2000	good
Metals Instrumentation	Thermo	ICAP 6000 Duo	ICP-20094603	2010	good
Metals Instrumentation	Thermo	ICAP 6000 Duo	ICP-20094602	2010	good
Water Quality Instrumentation	Metrohm	IC Model 881	4111	2013	good
Water Quality Instrumentation	Konelab	Aqua20	SEA032	2009	good
Water Quality Instrumentation	Flash Point Analyzer	HFP 339	Herzog	2007	good
Water Quality Instrumentation	OI	Carbon Analyzer Model 1030	A54TB0578P	2006	good
Water Quality Instrumentation	OI	Carbon Analyzer Model 1030	E616130020E	2006	good
Water Quality Instrumentation	Thermo	ECA 1200 TOX	2006 0373	2006	good
Water Quality Instrumentation	Horizon	Speed Vap	03-0415	2005	good
Water Quality Instrumentation	Konelab	20XT	E3719731	2005	good
Water Quality Instrumentation	Thermo	ECA 1200 TOX	2004 901	2004	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	20126	2004	good
Water Quality Instrumentation	Konelab	20	S5019455	2004	good
Water Quality Instrumentation	Glastron	CN Midi-distillation	2502	2003	good
Water Quality Instrumentation	Glastron	Phenol Midi-distillation	2069	2003	good
Water Quality Instrumentation	Glastron	Phenol Midi-distillation	2053	2003	good
Water Quality Instrumentation	Labtronics	BOD Autoanalyzer	270H3XB531	2004	good
Water Quality Instrumentation	Mantech	BOD Autoanalyzer	MT-184-216	2014	good
Water Quality Instrumentation	ManTech	PC Titrator	MS-OK2-607	2003	good
Water Quality Instrumentation	HACH	Spectrophotometer #DR/2500	30200004886	2003	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	2060196	2002	good

Water Quality Instrumentation	Spectronic	Genesis 4001/4	3SGC199091	2000	good
Water Quality Instrumentation	Lachat	Quickchem 8000 Autoanalyzer	A83000-1527	2000	good
Water Quality Instrumentation	Lachat	Quickchem 8500 Autoanalyzer	40300001665	2014	good
Water Quality Instrumentation	Lachat	Quickchem 8000 Autoanalyzer	A83000-1439	1999	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	99010157	1999	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	99110569	1999	good
Water Quality Instrumentation	BOD chamber		Revco	1994	good
Sample Preparation Equipment	CEM	Microwave MARS	MD3978	2013	good
Sample Preparation Equipment	Gilson	Fractionator Model GX-274	40579	2013	good
Sample Preparation Equipment	TurboVap	II	TV0529N12427	2006	good
Sample Preparation Equipment	TurboVap	II	TV0529N12428	2006	good
Sample Preparation Equipment	TurboVap	II	TV9445N5816	1996	good
Sample Preparation Equipment	TurboVap	II	TV9427N4133	1996	good
Sample Preparation Equipment	TurboVap	II	TV944N5819	1996	good
Sample Preparation Equipment	TurboVap	II	TV944N5820	1996	good
Sample Preparation Equipment	TurboVap	II	TV0024N9623	2000	good
Sample Preparation Equipment	TurboVap	II	TV0022N9604	2000	good
Sample Preparation Equipment	TurboVap	II	TV0312N11592	2003	good
Sample Preparation Equipment	TurboVap	II	TV0312N11591	2003	good
Sample Preparation Equipment	Organomation	Rot-X-Tractor	16902	1999	good
Sample Preparation Equipment	Organomation	Rot-X-Tractor	16907	1999	good
Sample Preparation Equipment	Organomation	Rot-X-Tractor	16913	1999	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G1647/C5659	1994	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2665/C5674	1994	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2620/C5660	1994	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2245/C6328	1995	good



Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2621/C6733	1995	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2713/C6732	1995	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G1643/C6837	1995	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G2742/C6842	1995	good

Table 20-2.

Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Change dryer tube Fill reductant bottle with 10% Stannous Chloride	Daily Daily As Needed Daily
ICP & ICP/MS	Check pump tubing Check liquid argon supply Check fluid level in waste container Check re-circulator levels Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Change pump oil Change Cones Change printer cartridge Replace pump tubing	Daily Daily Daily Monthly As required Daily Monthly Monthly Monthly As required As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly

Instrument	Procedure	Frequency
Agilent GC/MS	Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	Monthly Annually As required As required As required As required As required As required As required As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Glass wool replacement Check system for gas leaks with SNOOP Check for loose/frayed power wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required As required W/cylinder change as required As Required As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples and solvents Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required

Instrument	Procedure	Frequency
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
Centrifuge	Check brushes and bearings	Every 6 months or as needed

Table 20-3.

Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using "S" NIST traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	$\pm 0.2\%$	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using "S" NIST traceable. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	$\pm 0.5\%$	Clean. Replace.
NIST Certified Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST- Traceable Thermometer- Mercury	Accuracy determined by accredited measurement laboratory.	3 years	As per certificate.	Replace.
NIST- Traceable Thermometer- Digital	Accuracy determined by accredited measurement laboratory.	1 year	As per certificate	Replace.
Thermometer	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	$\pm 1.2^{\circ}\text{C}$	Replace
Minimum- Maximum Thermometers	Against NIST-traceable thermometer	Yearly	$\pm 1.5^{\circ}\text{C}$	Replace

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
InfraRed Temperature Guns	Against NIST-traceable thermometer Accuracy determined by accredited measurement laboratory.	Daily at appropriate temperature range for intended use. Annual	$\pm 1.5^{\circ}\text{C}$	Repair/replace
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	$\pm 1.5^{\circ}\text{C}$	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	$0-6^{\circ}\text{C}$	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	$(-10)-(-20)^{\circ}\text{C}$	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	$104 \pm 1^{\circ}\text{C}$ (drying) $180 \pm 2^{\circ}\text{C}$ (TDS)	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	$\pm 2^{\circ}\text{C}$	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water or solvent of use, dispense into tared vessel. Record weight with device ID number. Calibrate using 4 replicate gravimetric measurements	Each day of use Quarterly	$\pm 2\%$ Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Glass Microliter Syringes	None	Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy.	$\pm 1\%$	Not applicable.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Daily	$<1.0 \mu\text{mho}$ at 25°C	Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Manager.

SECTION 21

MEASUREMENT TRACEABILITY

21.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. For certain programs Microsyringes are verified semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g. bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory accreditation Cooperation) or APLAC (Asia – Pacific Laboratory Accreditation Cooperation)..A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to **TestAmerica Buffalo** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, "Standard Traceability and Preparation" and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory department's chemical history log and are assigned a unique identification number. Preparation of working standards or reagents prepared from the stock is documented in the laboratory Department's Standard Preparation Log. The following information is typically recorded:

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date

- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID from LIMS.
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the LIMS system.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

SECTION 22.0

SAMPLING

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

BF-FS-001	Chain of Custody Documentation
BF-FS-003	Groundwater Sampling Field Data Collection
BF-FS-004	Equipment Decontamination
BF-FS-005	Groundwater/Surface Water Sampling
BF-FS-006	Calibration of Field Meter
BF-FS-007	Low Flow Sampling Procedures
BF-FS-008	Surface and Subsurface Soil/Sediment Sampling

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory online.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in

“days” (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times, this info is in the SOP or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, “Sample Homogenization and Subsampling”.

SECTION 23

HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The shipping documents are retained with the project files.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

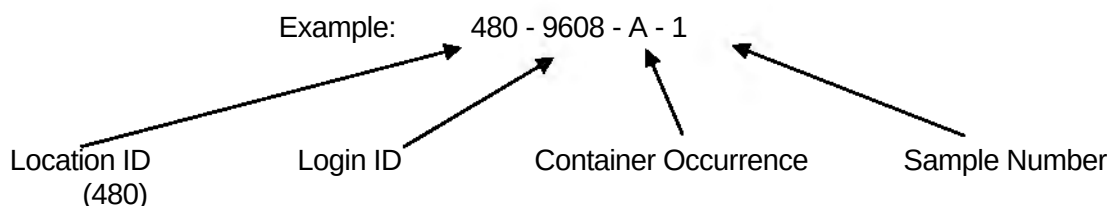
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Login Form – and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Buffalo Laboratory (Location 480). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - A ← Secondary Container Occurrence

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);

- sample holding times must be adhered to (Sampling Guide);
- every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.;
- The project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks described in Section 23.1.1.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. BF-SR-002.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis

the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel.

All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses (see Note), a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will analyze the trip blanks that were supplied.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record should be completed.

Figure 23-1.

Example: Chain of Custody (COC)

Chain of Custody Record										TestAmerica THE LEADER IN ENVIRONMENTAL TESTING									
TAL-4124 (1007) Client: _____ Project Manager: _____ Date: _____ Chain of Custody Number: 193232 Address: _____ Telephone Number (Area Code)/Fax Number: _____ Lab Number: _____ City: _____ State: _____ Zip Code: _____ Site Contact: _____ Lab Contact: _____ Project Name and Location (State): _____ Carrier/Waybill Number: _____ Contract/Purchase Order/Quote No.: _____ Matrix: _____ Containers & Preservatives: _____ Sample I.D. No. and Description (Containers for each sample may be combined on one line): _____ Date: _____ Time: _____ Analysis (Attach list if more space is needed): _____ Special Instructions/Conditions of Receipt: _____																			
Possible Hazard Identification ☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown Turn Around Time Required ☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other _____ Sample Disposal ☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month) QC Requirements (Specify): _____																			
1. Relinquished By: _____ Date: _____ Time: _____ 2. Relinquished By: _____ Date: _____ Time: _____ 3. Relinquished By: _____ Date: _____ Time: _____ Comments: _____																			
1. Received By: _____ Date: _____ Time: _____ 2. Received By: _____ Date: _____ Time: _____ 3. Received By: _____ Date: _____ Time: _____																			

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2.

Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

- 1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
 - *Client name, address, phone number and fax number (if available)*
 - *Project name and/or number*
 - *The sample identification*
 - *Date, time and location of sampling*
 - *The collectors name*
 - *The matrix description*
 - *The container description*
 - *The total number of each type of container*
 - *Preservatives used*
 - *Analysis requested*
 - *Requested turnaround time (TAT)*
 - *Any special instructions*
 - *Purchase Order number or billing information (e.g. quote number) if available*
 - *The date and time that each person received or relinquished the sample(s), including their signed name.*
 - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.***
 - **Information must be legible**
- 2) Every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.
- 3) Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or

courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

- 4) Samples must be properly labeled.
 - Use durable labels (labels provided by TestAmerica are preferred)
 - Include a unique identification number
 - Include sampling date and time & sampler ID
 - Include preservative used.
 - Use indelible ink
 - **Information must be legible**
- 5) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

- 6) Samples must be preserved according to the requirements of the requested analytical method. See lab Sampling Guide.

Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
- For Volatile Organic analyses in drinking water (Method 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
 - 1. Test for residual chlorine in the field prior to sampling.
 - If no chlorine is present, the samples are to be preserved using HCl as usual.
 - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
 - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE – for NPDES samples by Standard Methods or EPA 335**
 - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.
 - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements

or the laboratory can analyze the samples as delivered and qualify the results in the final report.

- It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
- The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

7) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.
 - Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for “field” analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.
- 8) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.
- 9) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

10) Recommendations for packing samples for shipment.

- Pack samples in Ice rather than “Blue” ice packs.
- Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.

Figure 23-3.
Example: Cooler Receipt Form

BF-SC-LF-003
Rev. 1
6/10/2013

SAMPLE LOGIN

Project _____ Event _____

Analysis Groups _____

TAT _____ # SAMPLES: _____ TRIP BLANK? Y/N _____ #/date _____

Custody Seal Intact Y/N NONE Rad Check <0.02 mR/hr Y/N

Residual Chlorine Check Y/N/ NA Pres Checked Y/N/NA

Workshare/Subcontract Y/N Lab _____ SO/ICOC # _____

Received out of hold: Samples _____ Analysis _____

Checklist/NCM's _____

Temperature(s)	#of coolers _____	IR Gun	1	2	3

RUSH
TIME CRITICAL

Section 24.0

ASSURING THE QUALITY OF TEST RESULTS

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1.

Control Type	Details
Method Blank (MB)	Are used to assess preparation and analysis for possible contamination during the preparation and processing steps. The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples. The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or quality associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Table 24-1.

Control Type	Details
Instrument Blanks	Are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank ¹	Are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan) Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks ¹	Are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks ¹	Are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

¹ When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

- 24.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix effects in a laboratory batch.
- 24.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.
- 24.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- 24.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- 24.4.1.5** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). In order to meet this requirement, TestAmerica Buffalo spikes with the Corporate Standard Standards primary mix for each analysis. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.
- 24.4.1.5.1** For methods that have 1-10 target analytes, spike all components.
- 24.4.1.5.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- 24.4.1.5.3** For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-5. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency ¹	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency ¹	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates ²	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency ¹	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency ¹	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

¹ See the specific analytical SOP for type and frequency of sample matrix control samples.

² LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.

24.6.3.4 The maximum acceptable recovery limit will be 150%.

24.6.3.5 The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by $< 5\%$ from previous, the data points are inspected and, using professional judgment, the limits may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. This process is outline in BF-QA-002.

24.6.4.1 The control limits are maintained in the laboratory LIMs system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMs system and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

24.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples.

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 19.

24.7.7 The laboratories sample acceptance policy is included in Section 23.

SECTION 25.0

REPORTING RESULTS

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. A variety of report formats are available to meet specific needs. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

The laboratory complies with any state reporting requirements. An example is located in BF-PM-008 – Massachusetts DEP Notification Procedures.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) with a "sample results" column header.

25.2.2 Each report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Practical quantitation limits or client reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the COC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.21 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g, partial report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as an addendum to the report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 Certification Summary report, where required, will document that unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

25.3 **REPORTING LEVEL OR REPORT TYPE**

TestAmerica Buffalo offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 26.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Buffalo offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. It is our policy that facsimiles are intended for and should be used for business purposes only. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this

communication is strictly prohibited. If you have received this communication in error, please notify the sender.

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The revised report will have the word "revised" appended to the cover letter.

When the report is re-issued, a notation of "revised" is placed on the cover/signature page of the report. A brief explanation of reason for the re-issue is included in the report case narrative.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

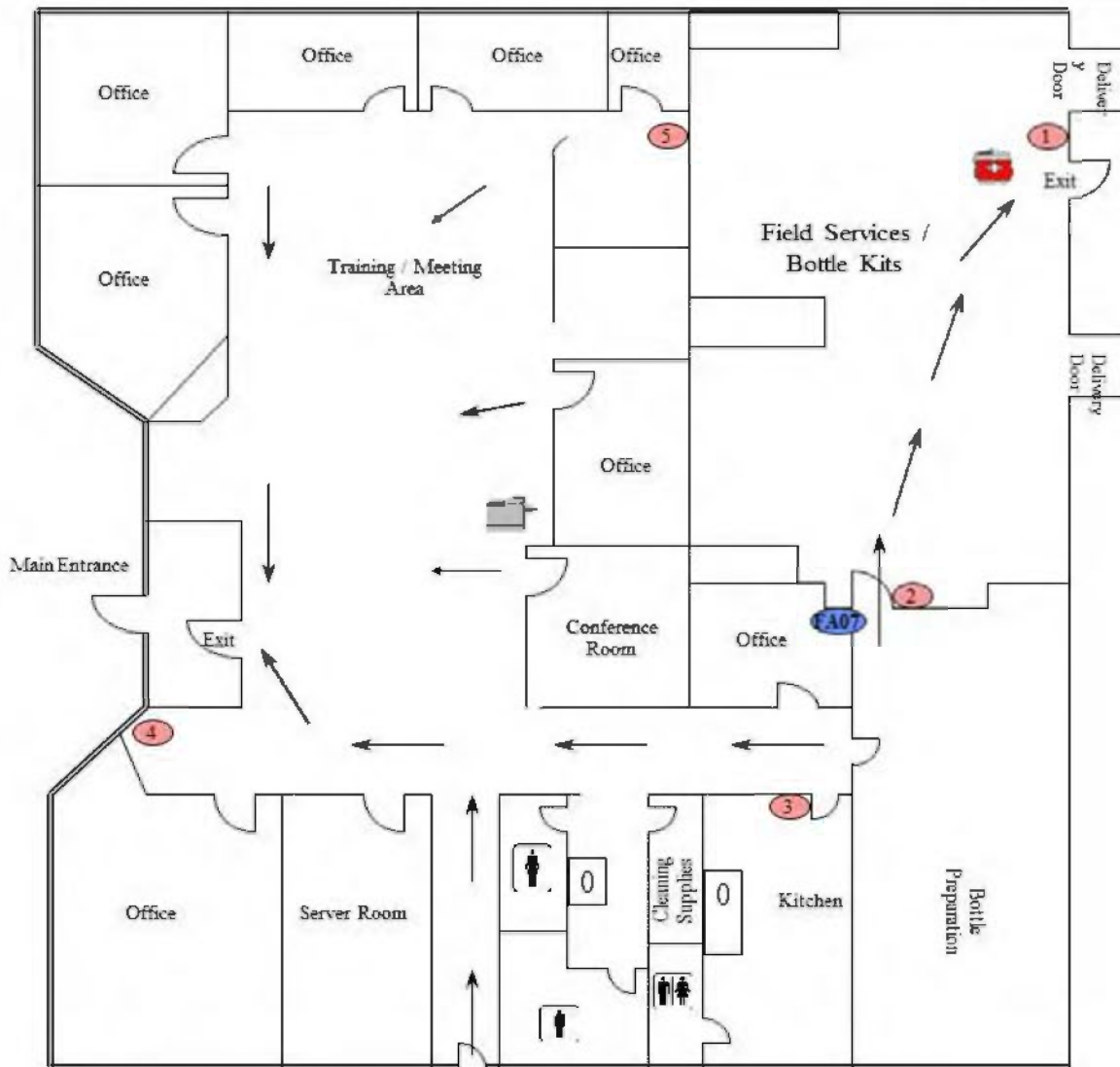
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1.

**TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 100
FLOOR PLAN**



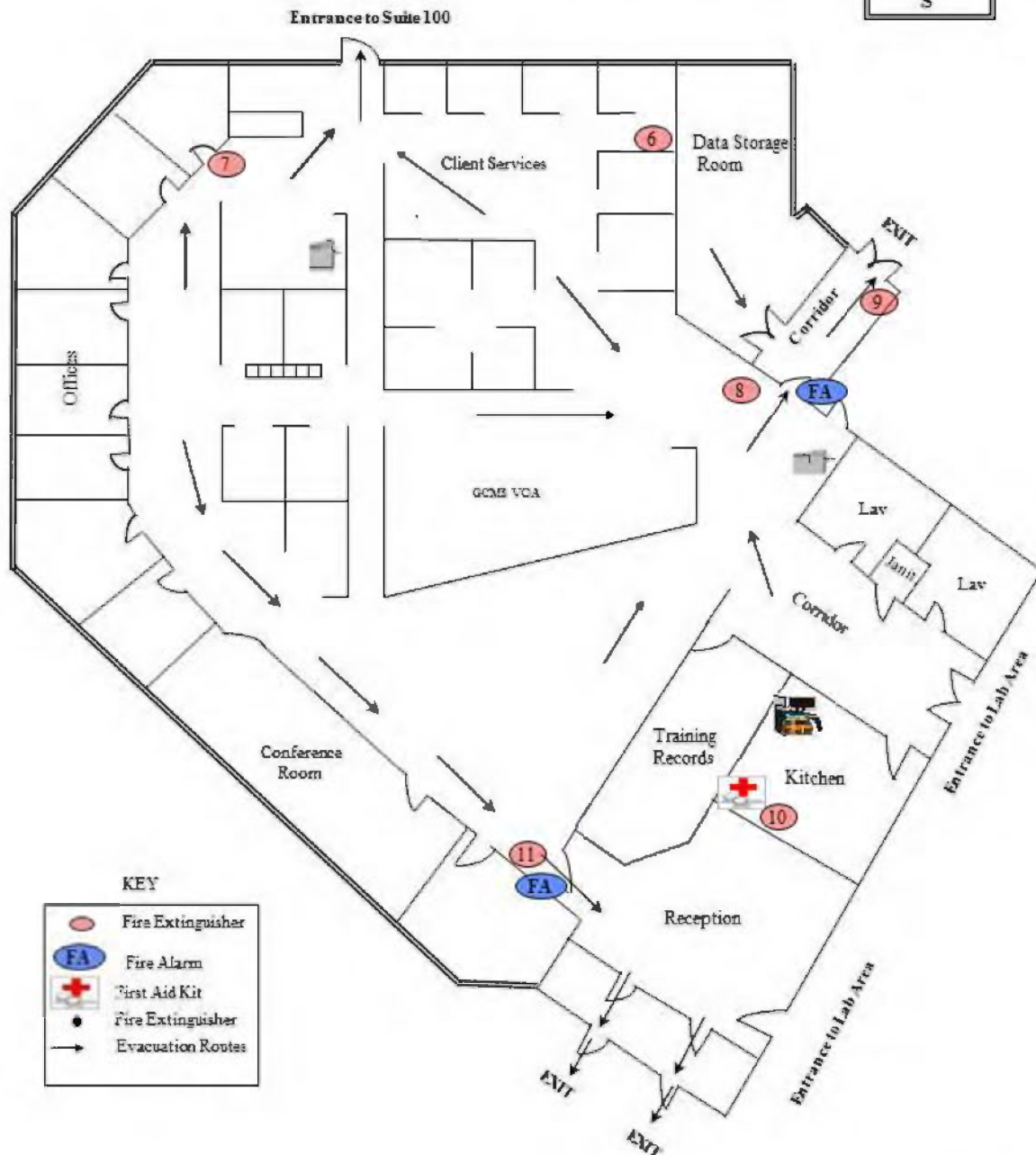
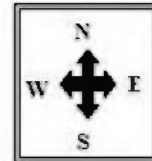
KEY



Doorway leading to Suite 106

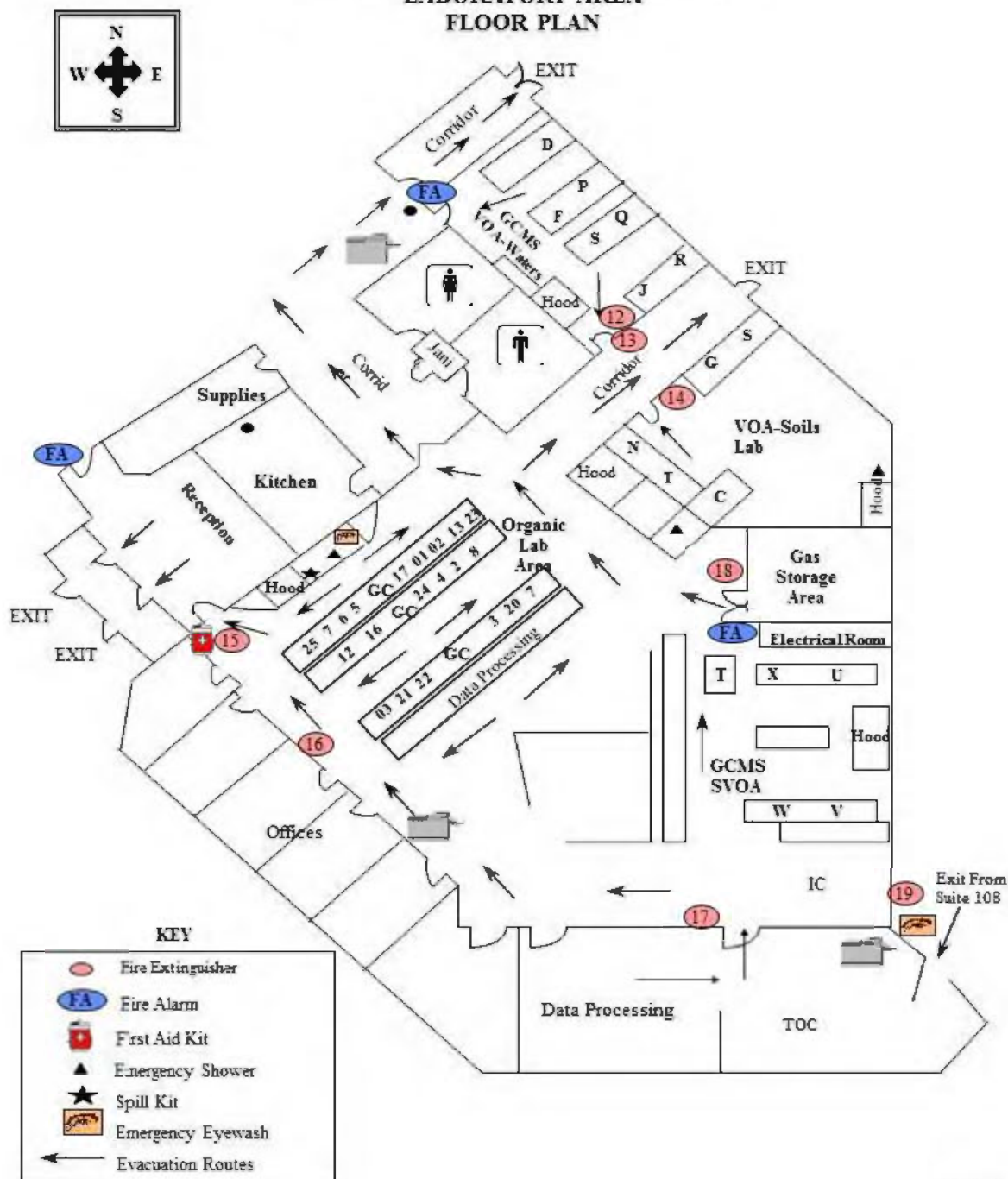
FrPn100

**TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 106
CLIENT SERVICES/REPORT PREP
FLOOR PLAN**



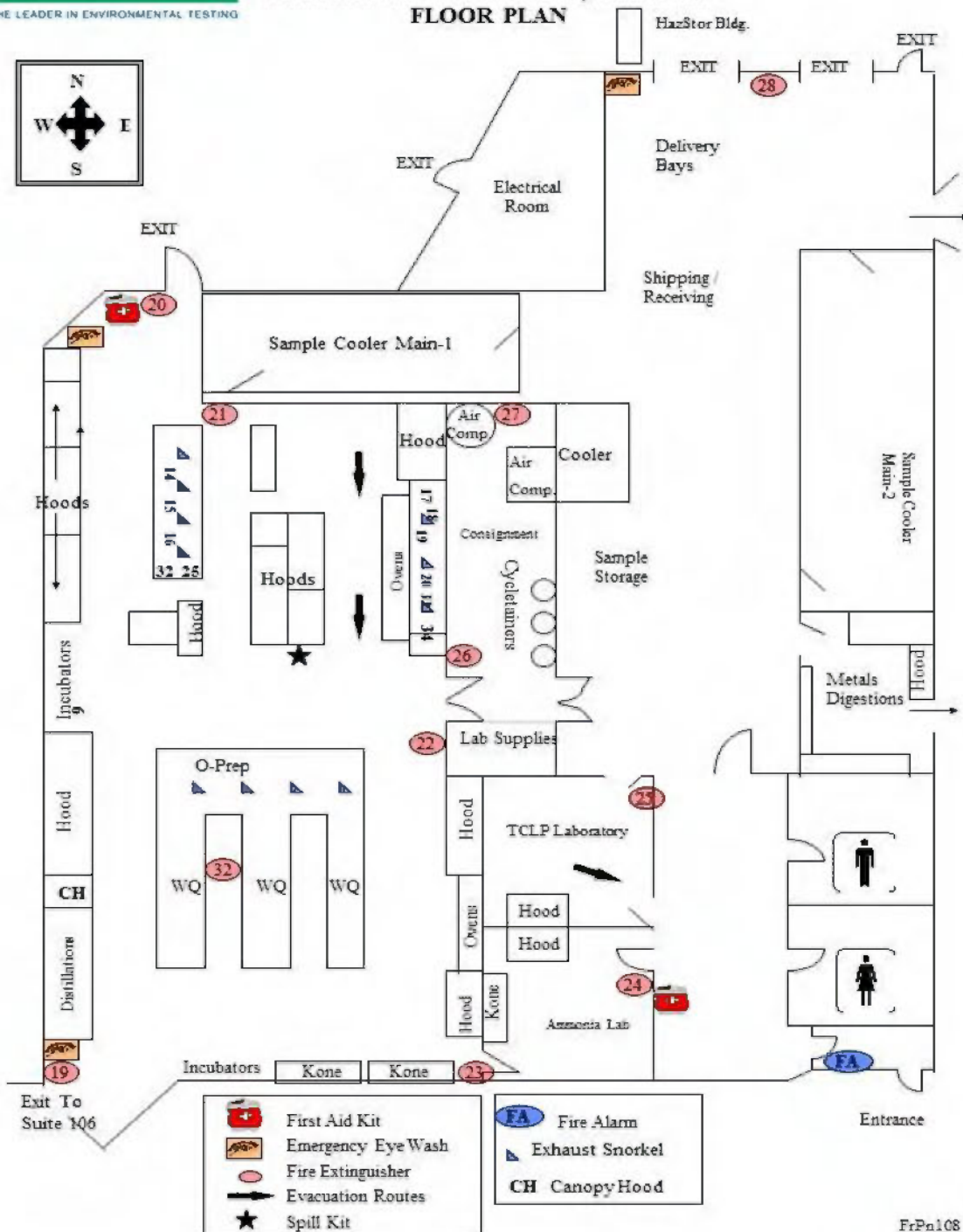
FrP1106L
3/2005

**TAL BUFFALO
HAZELWOOD DR. NY OFFICES, SUITE 106
LABORATORY AREA
FLOOR PLAN**

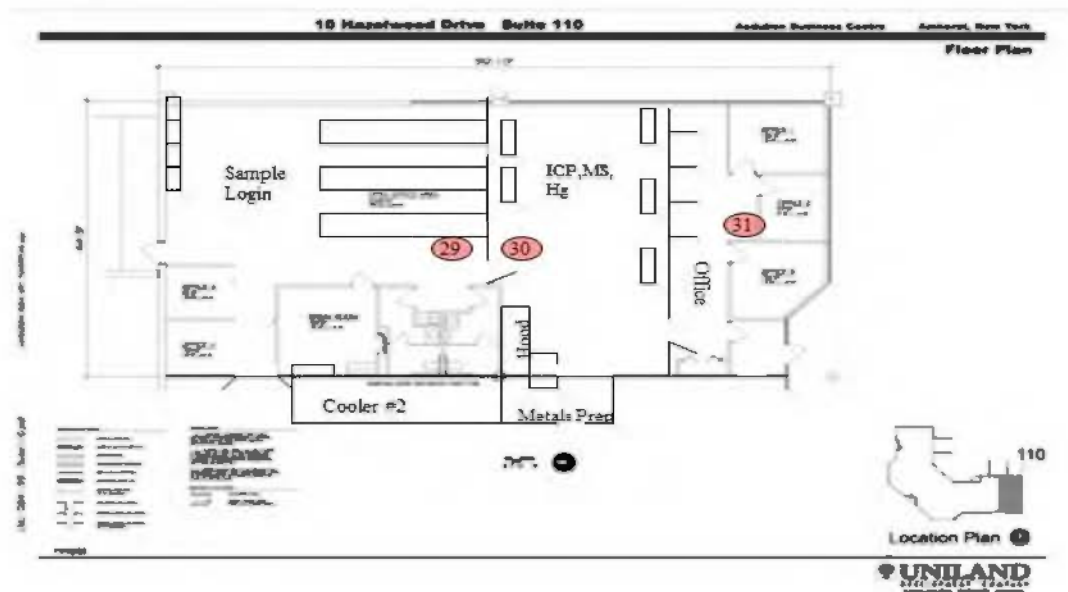


FrPn106r
03/2005

**TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 108
FLOOR PLAN**



FrPn108
03/2005



Appendix 2. Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

Accrediting Authority: The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory’s control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

- 1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
- 2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI).

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (TNI)

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional Cleanup procedures

(TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is $\pm 100\%$. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In

order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for Inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% Settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% Settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which

an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (TNI)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI) [2.1]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of NELAC standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or and which is accepted as the method for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not

available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS-ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate

LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
NELAC – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Buffalo maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

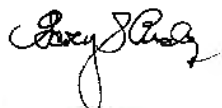
State	Program	Cert # / Lab ID
Arkansas	CWA, RCRA, SOIL	88-0686
California*	NELAP CWA, RCRA	01169CA
Connecticut	SDWA, CWA, RCRA, SOIL	PH-0568
Florida*	NELAP CWA, RCRA	E87672
Georgia*	SDWA, NELAP CWA, RCRA	956
Illinois*	NELAP SDWA, CWA, RCRA	200003
Iowa	SW/CS	374
Kansas*	NELAP SDWA, CWA, RCRA	E-10187
Kentucky	SDWA, CWA	90029
Kentucky UST	UST	30
Louisiana*	NELAP CWA, RCRA	2031
Maine	SDWA, CWA	NY0044
Maryland	SDWA	294
Massachusetts	SDWA, CWA	M-NY044
Michigan	SDWA	9937
Minnesota	SDWA, CWA, RCRA	036-999-337
New Hampshire Primary*	NELAP SDWA, CWA, RECRA	2973
New Hampshire Secondary*	NELAP SDWA, CWA, RECRA	2337
New Jersey*	NELAP, SDWA, CWA, RCRA,	NY455
New York*	NELAP, AIR, SDWA, CWA, RCRA	10026
North Dakota	CWA, RCRA	R-176
Oklahoma	CWA, RCRA	9421
Oregon*	CWA, RCRA	NY200003
Pennsylvania*	NELAP CWA, RCRA	68-00281
Rhode Island	SDWA, CWA	LAO00328
Tennessee	SDWA	02970
Texas*	NELAP CWA, RCRA	T104704412-08-TX
USDA	FOREIGN SOIL PERMIT	S-41579
Virginia	SDWA	278
Washington*	NELAP CWA, RCRA	C1677
Wisconsin	CWA, RCRA	998310390
West Virginia	CWA, RCRA	252

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

**Title: Chlorinated Herbicides
[Method 8151A]**

Once printed, this is considered an uncontrolled document.

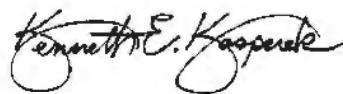
Approvals (Signature/Date):



Gary Rudz
Department Manager

9/29/14

Date



Kenneth Kasperek
Health & Safety Manager

9/29/14

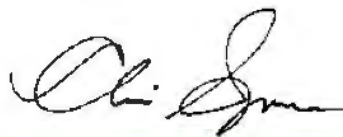
Date



Brad Prinzi
Quality Assurance Manager

9/29/14

Date



Christopher Spencer
Laboratory Director

9/29/14

Date

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1.0 Scope and Application

This method is used to qualify and quantify chlorinated acid herbicides by direct injection techniques into a capillary column equipped gas chromatograph. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the integration of gas chromatograms. Table 1 lists the compounds that may be analyzed using this method.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is used to determine chlorinated acid herbicides in soil and waters. See Table 1 for a listing of laboratory quantitation limits. These values are lab generated and reviewed annually.

Table 1

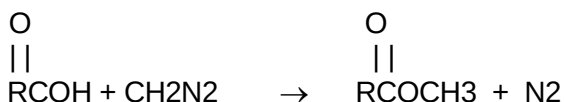
Analyte		CAS No.	Aqueous Laboratory RL (ug/L)	Solid Laboratory RL (ug/kg)
2,4-D Acid	2,4-Dichlorophenoxy Acetic	94-75-7	0.50	17
2,4,5-TP (Silvex)	2,4,5-Trichlorophenoxy Propionic Acid	93-72-1	0.50	17
2,4,5-T Acetic Acid	2,4,5-Trichlorophenoxy	93-76-5	0.50	17
Dalapon		75-99-0	0.50	17
Dichloroprop		120-36-5	0.50	17
Dinoseb		88-85-7	0.50	17
Pentachlorophenol		87-86-5	0.50	17
Picloram		1918-02-1	0.50	17

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.0 in the Quality Assurance Manual.

Additional analytes are being developed via a corporate initiative to standardize reporting lists in each lab; Dicamba, 2,4-DB, MCPA, & MCPP.

2.0 Summary of Method

Chlorinated Acid Herbicides are derivatized to form corresponding methyl esters with diazomethane.



These methylated compounds are then analyzed by Gas Chromatography on a HP5890-GC with an Electron Capture Detector using Helium as a carrier gas.

3.0 **Definitions**

- 3.1** 8151A MOD: The reference to a specific modified version of the general 8151A guidance method.
- 3.2** LCS: Synonym for laboratory quality control samples.
- 3.3** MS/MSD/SD: Synonyms for client sample spikes
- 3.4** DU/MD: Refer to duplicate samples
- 3.5** MB: refer to method blank
- 3.6** IBLK/HEXANE: Refers to instrument or solvent blanks used to identify contamination or minimize carryover problems
- 3.7** TEST RUNS: Used to evaluate instrument operational functionality
- 3.8** ICV: Initial 2nd source check for Calibration in TALS (Alt Source)
- 3.9** CCV: Continuing Calibration Verification
- 3.10** PRIMER: Old standard, QC, or blank analyzed after instrument has been idle
- 3.11** Derivitize: To convert a compound into another compound or structure that is chemically related to the original structure.
- 3.12** Methylation: To introduce a "methyl" group into a chemical or structure. The alkyl group: CH₃ is derived from Methane by the removal of a single Hydrogen atom.

4.0 **Interference**

- 4.1** Method interference can be minimized by proper glassware cleaning methods, as well as the use of high purity reagents and solvents.
- 4.2** Organic acids and phenols may cause interference in this method.
- 4.3** If heavily concentrated samples are encountered, contamination by carryover can occur. To avoid cross-contamination of subsequent samples, analysis of highly concentrated samples may be followed by the analysis of one or more blanks to verify instrument cleanliness.
- 4.4** If visual inspection of samples yields atypical appearance, filtration, dilution, or other procedure may be employed.

5.0 **Safety**

Employees must abide by the policies and procedures in the Corporate Safety Manual (CW – E-M-001) the facility addendum to the CSM, and this document. It is the responsibility of the analyst performing this method to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- 5.11** Nitrile, latex, and vinyl gloves all provide adequate protection against materials used in this method.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst need to be aware of the locations of these zones, and must cool them to room temperature prior to direct contact with them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, the power of the instrument may be turned off or may be disconnected from its power source.

5.2 Primary Materials Used

Table 2 contains a list of materials used in this method, which have serious or significant hazard rating. A complete list of the materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it and for the first time or when there are major changes to the MSDS.

Table 2

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hexane	Flammable, irritant	500 ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas Chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.
- Hewlett Packard 5890 Gas Chromatograph (or equivalent)

- Hewlett Packard 7673 Auto Sampler
- Hewlett-Packard 3396A Integrator
- Capillary columns as described in Section 6.2
- Electron Capture Detectors
- PE Nelson Totalchrom data system
- Carrier Gas – Helium
- Make-up Gas – Argon/Methane
- Syringes – various

6.1.1 Instrument Maintenance

- Upon Verification of established operating conditions the following may be performed either after failed QC or as preventative maintenance. All maintenance should be noted in the specific maintenance logbook.
 - Change Septum
 - Check flow and column conditions
 - Analyze solvent blank to check for contamination
 - If degradation or any interference is present, the analyst may replace the injection port liner, bake the column at 300°C or cut a section of the column end (i.e. 6-12 inches)

6.2 Supplies

Herbicides Dual Column Analytical Systems. A two-column system is required to provide confirmation of identification of the herbicides. The following supplies are recommended:

- Column Pair
- 30m x 0.32mm x 0.5µm RTXCLPI (or equivalent)
- 30m x 0.32mm x 0.5µm RTXCLPII (or equivalent)
- Restek Press-Tight “Y” Connector (Cat. #20404)
- Guard Column
- Column Configuration. The guard column is connected to the injection port. A “Y” connector is then placed at the end of the guard column to split the flow to the two analytical columns. These columns are then connected to two separate Electron Capture Detectors.

7.0 Recommended Reagents and Standards

- Certified Herbicide Mix (Ultra Scientific HBM-8151 A-1) or Supelco (46861-U)
- Herbicides Methyl Derivatives (EM Science EPM80138-1), (Ultra Scientific HBM-8151M) Alternate Source
- DCAA (surrogate) (2,4 Dichlorophenyl-acetic acid) (Restek 32049)
- DCAA Methyl ester solution (Ultra Scientific PPS-166-1)
- Additional vendor supplied standards as needed
- Hexane (reagent grade)
- Isooctane (reagent grade)
- Methanol (pesticide grade)
- (Trimethylsilyl) diazomethane solution
- Silicic Acid

8.0 Sample Collection, Preservation, Shipment and Storage

Both aqueous and soil samples must be stored and maintained at the lab at 4°C, ± 2 , in Teflon sealed amber containers until the analyst is prepared for the analytical process, which must take place no longer than 40 days after extraction. Table 3 details acceptable containers, preservation and holding times.

Table 3

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	Glass	1 L amber container	Cool 4 $\pm$ 2°C	7 days	N/A
Soils	Glass	4oz wide mouth	Cool 4 $\pm$ 2°C	28 days	N/A

9.0 Quality Control**9.1 Sample QC****Surrogates**

All samples, method blanks, blank spikes, and matrix spikes are fortified with the surrogate DCAA (2,4 Dichlorophenyl acetic acid) to monitor the performance of the extraction, methylation and, analytical analysis. Control limits are developed using procedures described in Standard Operating Procedure BF-QA-002; Quality Control Limits. If the values obtained are out of this range, re-extraction and re-analysis must be performed. If upon re-analysis the values are still outside the control limits, comments must be made describing the reason, i.e., matrix interference or chromatographic interference.

(Re-extraction for surrogate failure is not routinely performed for Leachate samples or TCLP samples due to the known sample pH effects on recoveries)

Method Blanks

A method blank is a volume of a clean reference matrix (reagent water for water samples) that is carried through the entire analytical procedure. The volume of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples. A method blank must be prepared and analyzed with each batch (maximum 20 samples). This method blank must contain a concentration less than the detection limit for all target analytes to be acceptable.

(For AFCEE and other DoD projects, the concentration in the method blank must be $<1/2$ the reporting limit.)

Lab Control Sample

A Lab Control Sample is a volume of a clean reference matrix spiked with a known quantity of target analytes and is carried through the entire analytical procedure. The volume of the reference matrix must be approximately equal to the volume or weight

of samples associated with the LCS. The LCS is analyzed in order to ensure that the analytical system is in control and to ensure that the laboratory is capable of making accurate and precise measurements. A Lab Control Sample Duplicate may be analyzed if there is insufficient volume for the analysis of a Matrix Spike and Matrix Spike Duplicate. Lab Control Sample and Lab Control Sample Duplicate recoveries should fall within the laboratory Quality Control limits that are determined using procedures described in Standard Operating Procedure BF-QA-002; Quality Control Limits. If the recoveries are not achieved, re-extraction and reanalysis must be performed.

If the recovery in the LCS is elevated and the associated samples are ND for the respective compound, the data are considered acceptable for reporting.

Matrix Spike/Matrix Spike Duplicate

A Matrix Spike and Matrix Spike Duplicate are an aliquot of an environmental sample spiked with known quantities of target analytes. The MS/MSD is carried through the entire analytical procedure and is evaluated to determine sample matrix bias on the analytical system or method. A MS/MSD shall be prepared once per each analytical batch or 1 per 20 environmental samples. If insufficient volume has been received for the analysis of a MS/MSD, a LCS/LCSD must be analyzed.

Table 4 summarizes the frequency and control limits of individual QC.

Table 4

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	Statistical Limits ⁴
Matrix Spike (MS) ²	1 in 20 or fewer samples	Statistical Limits ⁴
MS Duplicate (MSD) ²	1 in 20 or fewer samples	Statistical Limits ⁴
Surrogates	every sample ³	Statistical Limits ⁴

¹ LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

² The sample selected for MS/MSD are randomly chosen, unless specifically requested by a client.

³ Analytical and QC samples (MB, LCS, MS/MSD)

⁴ Statistical control limits are updated annually and are updated into LIMS.

9.2 ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

9.2.1 ICAL: %RSD < 20% or calibration factor >0.995

9.2.2 ICV (second source): Within +/-15% of true value

9.2.3 CCV: %D < 15%

If the CCV is out high and there are no positives in the samples the results may be reported. It must, however, be noted in the logbook and on the Job Summary.

9.2.4 Method Blank:

Detected concentrations < PQL

AFCEE or DoD requires conc.<1/2 RL.

Detected concentrations < 10X amount in associated samples

9.2.5 LCS:

Recovery within lab derived historical limits.

*DEP RPC limits must be within 30-150%

9.2.6 MS/MSD:

Recovery within lab derived historical limits.

9.2.7 Surrogate:

Recovery within lab historical limits.

*DEP RPC limits must be within 30-150%

9.3 Contingencies for Handling Unacceptable Data

A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

- Holding times exceeded
- Insufficient sample volume for re-extraction
- Re-extraction required due to method blank, surrogate or LCS failure

In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action.

For CCV's, a comment must be added to the report non-conformance summary stating which analytes exceeded the individual 15% D criteria.

10.0 Procedure

10.1 Standard Preparation

Preparation of Primary Herbicide/ Surrogate Intermediate Solution

Add 1ml isooctane, 0.5ml methanol and 1ml of hexane to a 10ml volumetric flask.

Accurately add 1.0ml of the certified herbicide mixture (from Ultra HBM-8151A) and 0.5ml of the DCAA solution (from Restek 32049) to the volumetric with a syringe.

Add 100ul (Trimethylsilyl) diazomethane solution and let it react for 1 hour. Make sure the yellow color persists. Quench the reaction with 0.1-0.2g silicic acid. Dilute to the 10mL mark with Hexane. Transfer the standard to a sealed amber vial, label and refrigerate until needed. (All components in this resulting solution are present at a concentration of 10µg/ml. This is the Herbicide/Surrogate Intermediate solution.

Additional sample preparation information can be located in the following:

- SOP #BP-OP-011

- SOP #BP-OP-012

10.1.1 GC Conditions

The following are recommended GC Conditions for model HP5890:

- Initial Temperature: 70°C hold for 0.5 min
- Rate1: 12°/minute to 210°C, hold 6 min
- Rate2: 20°/minute to 270°C, hold 4 min
- Inj A/B Temperature: 220°C
- Detector A/B Temperature: 330°C
- Epp B Pressure: 4.0psi @ 70°C – Const Flow Mode
- Injection volume: 1ul

Column			
RTX-CLPI		RTX-CLPII	
rt	compound	rt	compound
6.11	Dalapon	6.81	Dalapon
12.37	2,4-Dichlorophenylacetic acid	14.13	2,4-Dichlorophenylacetic acid
13.55	Dichloroprop	15.73	Dichloroprop
13.89	2,4-D	16.42	2,4-D
14.37	Pentachlorophenol	17.74	Pentachlorophenol
15.25	Silvex (2,4,5-TP)	18.55	Silvex (2,4,5-TP)
15.77	2,4,5-T	19.40	2,4,5-T
19.21	Picloram	20.78	Dinoseb
19.50	Dinoseb	22.13	Picloram

(Yellow highlighted components switch elution order on columns)

10.2 Calibration

A five-point calibration curve is recommended for each compound of interest. The following is a summary of the calibration concentration and the corresponding final concentration in sample extracts for each compound of interest.

10.2.1 Curve Preparation

The following describes the preparation of the Initial Calibration curve standards. (State specific requirements may require each calibration level to be derivitized separately) See section 11.2 for calibration curve calculations.

- 0.05 ng/μl Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 50μl of the Herbicide/surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.
- 0.10ng/μl Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 100μl of the Herbicide/surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.

- 0.25ng/μl Multicomponent Herbicide Mixed Solution and surrogate. This prepared by adding 250μl of the Herbicide/surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.
- 0.50ng/μl Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 500μl of the Herbicide/surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.
- 1.00ng/μl Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 1ml of the Herbicide/surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.
- 0.02ng/μl Multicomponent Herbicide mixed Solution is required for Pentachlorophenol calibration only. This is prepared by adding 20ul of the Herbicide/Surrogate intermediate solution to a 10ml volumetric flask and diluting to the mark with hexane.
- 0.01ng/μl Multicomponent Herbicide mixed Solution is required for Pentachlorophenol calibration only. This is prepared by adding 100μl of the 0.1ng/ul multicomponent herbicide mix (13.2.3) to a vial and adding 900ml of hexane.

The preparation of the 0.25ng/μl Alternate Source Multicomponent Herbicide Mixed solutions is detailed below.

0.25ng/μl Alternate Source Multicomponent Herbicide mix. This solution is prepared by adding 25μl of EM Science's Chlorinated Herbicides Methyl Derivatives (EPM 80138-1) and 25μl DCAA methyl ester solution (from Ultra Scientific) to a 10ml volumetric flask and diluting to the mark with hexane.

A purchased methyl ester standard may be employed also as a second source and as a reference to the laboratories derivatization process to verify responses.

Other mixes may be incorporated in order to provide additional information on compounds to be added to the routine list stated in this SOP, and to handle any variability in the second source provided by various vendors.

10.2.2 Curve Calibration

The %RSD can be calculated using equation 2 in section 11.2

- If the % RSD for all analytes is <20%, the curve can be assumed to be linear and the average Calibration Factor can be used to calculate the concentration of any compound detected during sample analysis.
- If the %RSD exceeds 20% for any compound the calibration can be verified using either linear regression.
- Linear regression may be used to calculate the concentrations of any compounds found in a sample, as long as that compound's correlation coefficient is >0.995.

- The alternative source described in section 10.2.1 may be used to verify the initial calibration.

10.2.3 Continuing Calibration

- The concentration of the standard used for calibration verification is prepared at the midpoint of the five-point initial curve.
- Analysis is performed after every 10 samples and at the end of a particular sequence of samples.
- The calibration factor of the calibrator verification standard must be $\pm 15\%$ Difference (D) of the average calibration factor of the initial calibration curve. Failure to meet the 15% D criteria for calibration verification requires maintenance. If the maintenance performed is insufficient, an analyst may recalibrate the instrument (new initial curve).

Alternate source – A midpoint standard must also be prepared using a solution of the herbicide mixture of interest from a second source. This provides a check of our calibration curve and methylation procedure. This alternate source must be run after a new calibration curve and must have a %D of $\pm 20\%$ of the average response factor of the curve.

Table three summarizes the parameters for Calibration.

Table 3

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	$\leq 20\%$ RSD or $R^2 > 0.990$
Continuing Calibration Verification (CCV)	Prior to / after every 10 injections	$< 15\%$ D
RT Windows (RTW)	Init. CCV determines mid pt. of RTW	$\pm 3X$ SD

10.3 Sample Analysis

Samples are run on an HP5890 gas chromatograph using Hexane as a solvent and Helium as a carrier gas. Table 4 shows an example analysis queue / sequence for method 8151.

Table 4

Step	Standards	Type	Control Limit	Frequency
Method # 8151				
Initial Cal	5 to 7pt	Linear or Ave Cal Factor	$0.990 r^2$ or 20% RSD	As Needed
ICV	2 nd Source	Lot or Vender	$< 20\%$ D	After Every ICAL

CCV	ICAL Std	~ Midpoint	< 20% D	Prior to analysis
10 Runs	MBLK	Blank		Every 20 Samples
	LCS	Spike	In-House Limits	Every 20 Samples
	LCSD	Spike	In-House Limits	Every 20 Samples if no MS/MSD
	MSMSD	Sample Spike		As required by clients
	Samples & Inst Blanks			
CCV	ICAL Std	~ Midpoint	< 20% D	Every 10 Runs
10 Runs	Samples & Inst Blanks			
CCV	ICAL Std	~ Midpoint	< 20% D	Every 10 Runs

10.4 Retention Time Windows

The width of the retention time window for each compound is determined using the standard deviation of three retention time measurements of individual standards measured over the course of a 72-hour period.

- Make three injections of a mid-level standard mixture throughout a 72-hour period. Retention time windows are defined as plus or minus three times the standard deviation of the absolute retention time for each component. The experience of the analyst should weigh heavily in the interpretation of the chromatogram.

Retention time windows are to be calculated for each standard on each GC column on an annual basis or whenever the chromatographic conditions change, (i.e., a new GC column is installed or the instrument is subjected to major maintenance.) If the calculated windows encompass a variable range of 0.0 min to 0.10 minutes, a default of 0.05 minutes can be applied to all for consistency.

When relative retention time windows are updated, the new calibration method will be printed out for review of proper elution order for the particular column, and then dated and initialed upon completion of review. This will be filed with the initial curve for comparison.

11.0 Calculations / Data Reduction

11.1 CF Calculation Details

The concentration of specific analyses in samples is determined using the Average Calibration Factor (CF) calculated from the initial curve.

Equation 1: $CF = \text{Peak Area} / \text{Standard Concentration (ng/}\mu\text{l)}$.

Aqueous Calculation

Determine the calibration factor for each compound in the initial curve using equation 1 above, and calculate the ACF.

NG of Compound in Sample = Peak Area of Compound/ACF

$$\mu\text{g/l of Compound} = \{[\text{NG} \times \text{DF} \times \text{FV}] / [\text{I,V} \times \text{ExV}]\}$$

DF = Dilution Factor

FV = Final Volume of Concentrated Extract (mls)

I,V = Injection Volume (μls)

ExV = Extraction Volume (L)

Soil/Solid Calculation

Determine the calibration factor for each compound in the initial curve using equation 1 above, and then calculate the ACF.

NG of compound in Sample = Peak Area of Compound/ACF.

$$\mu\text{g/KG of Compound} = \{[\text{NG} \times \text{DF} \times \text{FV}] / [\text{I,V} \times \text{ExW} \times \text{DW}]\} \times 1000$$

DF = Dilution Factor

FV = Final Volume of Concentrated Extract (mls)

I,V = Injection Volume (μls)

ExW = Extraction Weight (grams)

DW = Decimal Dry Weight

11.2 Calibration Calculations

Equation 2: %RSD= [CF Standard Deviation/CF Mean] X 100

Equation 3: % D = [Avg.CF (Initial) – CF (Cal. Verif.)/Avg. CF (Initial)] X 10

- 11.3** Through the use of a Totalchrom data link, the nanogram amounts from the quantitation system are downloaded into the TALS system, where the final calculation is performed electronically. A second analyst reviews this data.

12.0 METHOD PERFORMANCE

12.1 Method Detection Limit

A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See SOP AQA-BF-QA-001, "The Determination of Method Detection Limits," current revision, for further guidance. Current Test America MDLs are maintained by the QA department and are easily viewed in the TALS.

12.2 Demonstration of Capabilities

A one-time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

This requires quadruplicate analysis of a mid-level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.0 Pollution Control

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

14.0 Waste Management

Waste Management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes are disposed of in an acceptable manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

The following waste types are generated for this procedure and where they are to be disposed of:

- C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards (except PCB's) will also be dumped into C-Waste.
- Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.
- Glass: contaminated glass needs to be rinsed with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.
- Extract Vials: extract vials are to be archived after they have been shot. After one year of archival vials are to be crushed into a 55-gallon drum.
- All acidified aqueous waste is to be disposed into a labeled "A" waste container.
-

15.0 References / Cross-References

Method 8151A U.S. Environmental Protection Agency, Office of Solid Waste and Energy Response, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods" 3rd Ed.,

SW-846; update III, Dec. 96.

16.0 Method Modifications: N/A

17.0 Dual Column Procedure

17.1 Dual Column Procedure (Policy No. CA-Y-P-003)

17.1.1 The laboratory may designate a primary column for each analyte (in which case the 2nd column is the confirmation column for each analyte), or may the lower result from the two columns. The same column does not have to be primary for every analyte. Both columns are intended to be calibrated and meet verification requirements.

17.1.2 Whichever approach is used QC must be assessed and reported in the same manner as the samples (i.e. report from a primary column or the lower number)

17.1.3 40% RPD Criterion

17.1.3.1 When using the primary column option, if the RPD for the dual column results is $\leq 40\%$, then the result from the primary column is normally reported. The exception is situations where a chromatographic interferent is evident in the primary column chromatogram that is not present in the confirmatory column chromatogram.

17.1.3.2 If using the primary column option and the RPD for the dual column results is $>40\%$, then the lower value is reported. (An RPD of $>40\%$ is clear evidence of chromatographic interference/co-elution on the higher value) A case narrative comment, flag, or footnote citing this failure must be included in the final report.

17.1.4 Calibration Verification Criterion – Primary Column Option

17.1.4.1 If the CCV recovery for the primary column is within acceptance limits and the analyte is confirmed on the secondary column, but the second column is outside of acceptance limits, then the primary column may be reported with a case narrative comment or footnote.

17.1.4.2 If the CCV recovery confirmation column is within acceptance limits, and the recovery for the primary column is in the range of +15 to +40%, then the results may be reported from the confirmation column with a case narrative comment or footnote.

17.1.4.3 If the CCV recovery is below acceptance limits for the primary column, or the secondary column is below acceptance limits and the analyte is not confirmed, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed with acceptable CCVs.

17.1.4.4 If the CCV recovery on either column is $>40\%$ above acceptance limits, any associated sample positives will need to be reanalyzed with acceptable CCVs.

17.1.5 Calibration Verification Criterion – Lower Result Option

17.1.5.1 If the CCV recovery for the column with the lower result is within acceptance limits and the analyte is confirmed on the secondary column, but the secondary column is outside acceptance limits ,

then results from the lower column may be reported with a case narrative comment or footnote.

17.1.5.2 If the CCV recovery for the higher result is within acceptance limits, but the lower results column fails acceptance criteria and the RPD is <15%, then the higher result may be reported with a case narrative comment or footnote.

17.1.5.3 If the CCV recovery on either column is >40% above acceptance limits, any associated sample positives will need to be reanalyzed with acceptable CCVs.

18.0 Revision History

Revision 0, dated 27 October 2006

- o Deleted Calcium Carbonate Powder from section 10.1
- o Deleted reference to Calcium Carbonate and sample prep section 14.1 and renumbered section.
- o Removed Dicamba from ICAL summary table under section 14.2.1

Revision 1, dated 01 August 2009

- o Revised into new format and revision, removed all Grand Mean references.

Revision 2, dated 07, September 2011

- o Revised to reference TALS nomenclature, removed ELEMENT references in many sections.
- o Section 3.0, Corrected 8151A reference
- o Section 1.0, reformatted Table 1.
- o Changed Quality Manager, signature added.

Revision 3, dated 13, July 2012

- o Section 9.2, Added DEP RCP requirements.

Revision 4, dated 1, April 2014

- o Section 1.1, added additional compounds targeted via the standard standard corporate directive.
- o Section 7.0, expanded reagent list as per above.
- o Section 10.2.1, expanded options for curve preparation to encompass additional compound development.
- o Update Quality Manager

Revision 5, dated 29, September 2014

- o Section 17 Inserted Dual Column procedure cited from Policy No. CA-Y-P-003
- o Section 10.1 Updated instrument conditions
- o Section 10.1 Inserted elution order and relative RT time table
- o Section 9.2.8 removed old dual column approach

**Title: Organochloride Pesticides
[Methods 608, 8081A, & 8081B]**

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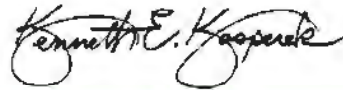
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1.0 **Scope and Application**

- 1.1 This method is used to qualify and quantify organochlorine Pesticides in extracts from solid and liquid matrices by direct injection techniques onto a capillary column. An electron capture detector (ECD) is employed for identification and quantification.
- 1.2 Pesticides are analyzed in an analytical run to allow separation of all single component pesticides.
- 1.3 The presence of single component pesticides is confirmed with a second dissimilar column and is based on retention time, peak shape, and response.
- 1.4 Pesticides analyses are performed in a single injection with a "Y" split into two analytical column systems.
- 1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the interpretation of gas chromatograms. Table 4 indicates compounds that may be determined by this method.

1.6 **Analytes, Matrix(s), and Reporting Limits**

- 1.6.1 Applicable matrices are soil or aqueous samples
- 1.6.2 Reporting limits are defined on an analyte specific basis by the low point in the calibration curve and are supported by the performance of annual method detection limit studies

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 **Summary of Method**

- 2.1 Soil or solid samples are extracted with an appropriate solvent using ultrasonic extraction (SOP BF-OP-005). For aqueous samples, approximately 1 liter of sample is extracted with methylene chloride using a separatory funnel (SOP BF-OP-003). The extract is then exchanged to hexane and concentrated to 10 ml or less. The final extract is then separated by gas chromatography and detected by an electron capture detector. Florisil column cleanup procedures and sulfur removal procedures may be utilized to mitigate any interferences that may be encountered during analysis. Although these procedures may eliminate several interferences, contamination of the sample may come from a variety of sources, including solvents, reagents, glassware and any of the hardware used in sample processing. For this reason, reagent and solvent blanks shall be analyzed to insure their purity.

3.0 **Definitions**

3.1 MDL

The theoretical calculated method detection limit using statistical means.

3.2 PQL

The practical quantification limit based on the lowest level of the calibration curve.

3.3 CRQL /CDL

The client required quantification limit or the client detection limit based on specific client or site regulations

3.4 Multi-component

The target compounds that exhibit many peaks as individual parts of the reported component. e.g. Toxaphene & Chlordane (also called technical chlordane)

3.5 LCS

Laboratory Quality Control samples.

3.6 MS/MSD/SD

Synonyms for client spikes

3.7 DU/MD

refers to duplicate samples

3.8 VBLK/MBLK/BLK

refer to Method Blanks

3.9 IBLK/HEXANE

refer to instrument or solvents used to identify contamination or minimize carry over problems

3.10 EBLK/BLK2

refers to the extractor blank performed with TCLP tumbling in TALs.

3.11 TEST RUNS

used to evaluate instrument operational functionality

3.12 ICV

Initial Calibration Verification-2nd source check for calibration in TALs (Alt Source)

3.13 IS

Internal Standard: 1-bromo-2-nitrobenzene

3.13 CCV

Continuing Calibration Verification

3.14 Primer

Old standard, QC, or blank analyzed if instrument has been idle

4.0 Interferences

4.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which ECD will respond. Interferences co-extracted from the samples will vary considerably. As part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination.

4.2 Gel Permeation Cleanup (SOP BF-OP-009 3460A) and Florisil Cartridge Cleanup (SOP BF-OP-007 3610A) for Pesticides are not routinely performed unless specifically required by a project.

5.0 Safety

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

5.1 Specific Safety Concerns or Requirements

5.1.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheets (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Gas chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.

6.1.1 Hewlett Packard 5890 gas chromatograph/Agilent 6890 gas chromatograph

6.1.2 Hewlett Packard 7673 Auto Sampler/Agilent 7683 Auto Sampler

6.1.3 Hewlett Packard 3392 and 3396A Integrators for HP5890 GC only

6.1.4 Capillary columns as described in sections 6.2

6.1.5 Electron Capture Detector

6.1.6 PE Nelson Turbochrom data system

6.1.7 Carrier Gas – Helium / Hydrogen

6.1.8 Make Up Gas - Argon/Methane / Nitrogen

6.1.9 Syringes – various

6.1.10 CHROM – TALs chromatography system

6.2 Pesticides dual column analytical system

6.2.1 Column pair or equivalents

6.2.1.1 Dual column injection Tee (Restek Corporation) 1/4 inch glass injection tee, deactivated.

6.2.1.2 30 m x 0.53 mm ID RTX-CLPI (Restek).

6.2.1.3 30 m x 0.53 mm ID RTX-CLPII (Restek)

6.2.1.4 30 m x 0.32 mm ID RTX-CLPI (Restek)

6.2.1.5 30 m x 0.32 mm ID RTX-CLPI (Restek)

6.3 Instrument Maintenance

6.3.1 Upon verification of established operating conditions, the following is performed on a sequence basis, or sooner if deemed necessary. All maintenance shall be noted in the specific maintenance logbook.

6.3.1.1 Change septum

6.3.1.2 Check flows and column condition (i.e., degradation)

6.3.1.3 Analyze solvent blank to insure system is free of contamination

6.3.1.4 If degradation or any interferences are present, the analyst may replace the injection port liner, bake the column at 300°C or cut a section of the column end (i.e., 6-12 inches).

6.3.1.5 Cut guard and/ or column

6.4 Supplies

- Volumetric Flasks (Class A): 100 mLs; 200 mLs; 500 mLs; 1000 mLs
- Syringes Various sizes

7.0 Reagents and Standards

7.1 Pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

7.1.1 Standards are stored in the GC Standard Incubator at $4 \pm 2^\circ\text{C}$ in teflon-sealed amber containers.

7.1.2 All stock standard solutions are replaced before the expiration date. All other standard dilutions or working standards are discarded after six month (or at the stock standard expiration date, whichever comes first) or sooner if routine QC indicates a problem.

7.1.3 All standards (Primary and Alternate Source) shall be acquired from a certified vendor. For standard traceability and preparation, refer to SOP BF-GP-019.

7.1.4 Calibration Standards for Pesticides

7.1.4.1 1-Bromo-2-Nitrobenzene: Stock 100 ng/ul, to be added ultimately to each extract and standard at a final concentration of 0.5ng/ul (ug/ml)

7.1.4.2 Intermediate Surrogate Mix Solution: 200 ng\ul Tetrachloro-m-xylene and Decachlorobiphenyl surrogate stock solution is diluted by 10 in Hexane to make a 20.0 ng\ul intermediate solution. Dilutions are prepared at five concentrations. (0.005 ng\ul, 0.010 ng\ul, 0.05 ng\ul, 0.075 ng\ul and 0.10 ng\ul).

7.1.4.3 Intermediate Single Component Pesticides Mix Solution: 1,000 ng\ul stock standard solution is diluted by 100 in Hexane to make the 10.0 ng\ul intermediate standard. The intermediate standard is subsequently diluted to prepare five concentrations (0.005 ng\ul, 0.010 ng\ul, 0.05 ng\ul, 0.10 ng\ul and 0.15 ng\ul).

7.1.4.4 The intermediate solution can be made from a stock containing both pesticides and surrogates at 1000ng/ul diluted by 100 in Hexane to make the 10.0 ng\ul intermediate standard. The intermediate standard and the intermediate surrogate solution are respectively and subsequently diluted to prepare five concentrations (0.005 ng\ul, 0.010 ng\ul, 0.05 ng\ul, 0.10 ng\ul and 0.15 ng\ul).

7.1.4.5 Intermediate multi-component Pesticides Mix Solution: The 200 ng/ul stock standard solution and the intermediate surrogate solution are respectively and subsequently diluted to prepare five concentrations (0.050 ng\ul, 0.10 ng\ul, 0.500 ng\ul, 1.000 ng\ul and 1.500 ng\ul).

7.1.4.6 Performance Evaluation Mix (PEM): The PEM stock solution is made of alpha-BHC, beta-BHC and gamma-BHC at 1.0 ng/μl of each pesticide, Endrin at 5.0 ng/μl, DDT at 10.0 ng/μl and Methoxychlor at 25 ng/μl. The intermediate PEM solution is prepared by adding 100 μl of the stock PEM solution to a 10.0 ml volumetric flask and bringing to volume with Hexane.

7.1.5 Spike Solution

7.1.5.1 The spike mixture contains each single-component parameter of interest at 1.0 ng/μl in acetone or methanol. For all routine analysis, 500ul of the spiking solution will be added to each water/soil spike (extract final volume = 10.0ml). The applicable expected concentration in the extract for all single component pesticides is then 0.050ng/ul. This concentration is equal to the midpoint of the calibration curve, and below the midrange of the calibration curve range. Additional information on the spike mixture can be found in the sample preparation SOPs BF-OP-005, and BF-OP-003.

7.1.5.2 For 3510C_LVI, the final volume of 2ml will be applied to utilize a 250ml extraction volume, and maintain the same reporting limits as a 1 Liter extraction.

7.2 See Table 1 for Summary Concentrations

8.0 Sample Collection, Preservation, Shipment and Storage

- 8.1** Aqueous samples are to be collected in a 1-liter amber glass jar and stored at 4±2°C. Organic preparation is to be performed within 7 days of collection. Analysis of the extracts is to be performed within 40 days of preparation.
- 8.2** Soil samples are collected in a 2 or 4 oz wide mouth glass jar and stored at 4±2°C. Organic preparation is to be performed within 14 days of collection. Analysis of the extracts is to be performed within 40 days of preparation.
- 8.3** Holding times specified in project specific quality assurance plans may supercede the above listed method criteria.
- 8.4** Extracts are stored under refrigeration at 4±2°C.
- 8.5** Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	1-Liter Amber glass	1000 mLs	Cool 4 ± 2°C	7 Days to extraction; 40 days to analysis	40 CFR Part 136.3
Soils	2 or 4oz wide mouth glass jar	30 grams	Cool 4 ± 2°C	14 Days to extraction; 40 days to analysis	N/A

9.0 Quality Control

9.1 The reader is referred to TestAmerica Laboratories' Corporate Quality Management Plan (QMP) and TestAmerica's Laboratory Quality Manual (LQM) for general information and more specific detail. Often project-specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than TestAmerica Laboratories' QMP, LQM or the following criteria. The following criteria are subsequently presented as minimum criteria or those criteria deemed applicable in the absence of project-specific DQOs.

9.2 Sample QC -The following quality control samples are prepared with each batch of samples.

9.2.1 Surrogates:

9.2.1.1 Lab QC and sample surrogate recoveries shall fall within method, lab calculated, or client specific limits. Samples outside these ranges are to be reanalyzed. Blank recoveries outside this range may lead to: Re-extraction if within holding time and volume available, noting recoveries in case narrative or flagging values as estimated. The spike results, sample matrix and reported positives in the prep batch are also to be considered. Acceptability may be determined by citing historical sample and method results on a case by case basis.

9.2.2 Matrix Spike/Matrix Spike Duplicates (MS/MSD)/Method Blank (LCS):

9.2.2.1 MS/MSD samples are to be prepared at a frequency of at least 5% (1 MS/20 samples and 1 MSD/20 samples) or with each analytical batch when sample volume permits it. A LCS is also to be prepared at a frequency of at least 5% (1 LCS /20 samples) or with each analytical batch. If insufficient field sample volume is available for MS/MSD in a batch of 20 samples, a LCS/LCSD shall be prepared. (For method 608, the spike frequency is 1 MS/10 Samples)

9.2.2.2 The percent recovery of the MS/MSD/LCS is calculated to determine the accuracy (%R) and precision (RPD) acceptability of the extraction batch. The laboratory's internal QA/QC limits are calculated annually and are used to monitor the laboratory quality performance and/or to identify trends in analysis. Laboratory limits are reviewed to determine compliance with the method acceptance criteria.

$$\%R = \{ [\text{Conc. spiked sample} - \text{Conc. base sample}] / \text{Spike added} \} \times 100$$

$$\text{RPD} = [\text{MS} - \text{MSD}] / \{[\text{MS} + \text{MSD}] / 2\} \times 100$$

9.2.2.3 If both the MS and MSD recovery fall outside the established control limits, matrix interference is assumed and LCS recovery acceptable, no corrective action is performed.

9.2.2.4 If only the MS or MSD falls outside the established control limits, the system shall be investigated to determine a possible cause of error.

9.2.2.5 If the recovery of the LCS falls outside control limits, the system shall be investigated to determine a possible cause of error. Re-extraction/re-analysis of the entire extraction batch may be performed. Lab QC recoveries shall fall within method, lab calculated, or client specific limits. QC outside these ranges are to be reanalyzed. Recoveries outside this range may lead to: Re-extraction if within holding time and volume available, noting recoveries in case narrative or flagging values as estimated. The spike results, sample matrix and reported positives in the prep batch are also to be considered. Acceptability may be determined by citing historical sample and method results on a case by case basis.

9.2.3 QC acceptance criteria for MS samples and LCS

9.2.3.1 Where in-house limits have been developed for matrix spike recoveries, the LCS results shall fall within those limits, as the LCS is prepared in a clean matrix. The laboratory shall use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. It is necessary for laboratories to develop in-house performance criteria and compare them to those in the methods. In-house limits are developed with a minimum of 15-20 spike results set by the formula.

9.2.3.2 For each matrix spike blank sample analyzed, calculate the percent recovery of each spike compound added to the blank. For each field sample, calculate the percent recovery of each surrogate.

9.2.3.3 Calculate the average percent recovery (p) and the standard deviation (s) for each of the spike compounds after analysis of 15-20 matrix spike blanks of the same matrix, using the equations below, as guidance. Calculate the average percent recovery (p) and the standard deviation (s) for each of the surrogates after analysis of 15-20 field samples of the same matrix, in a similar fashion.

9.2.3.4 After the analysis of 15-20 matrix spike blanks of a particular matrix (for matrix spike limits) or 15-20 field samples (for surrogate limits), calculate upper and lower control limits for each spike or surrogate compound:

Upper control limit = $p + 3s$

Lower control limit = $p - 3s$

9.2.3.5 The laboratory will develop and retain in-house limits to be evaluated annually based on historical results.

9.3 Method Blank

9.3.1 At a minimum, one method blank per extraction batch must be extracted and analyzed. (Minimum one/20 Samples). The concentration of any target compound in the method blank must be less than the reporting limit.

- 9.3.2** If the concentration of any compound exceeds the reporting limit, the system shall be investigated to determine a possible cause of error. If there are no positives in the associated samples, the data may be used noting the exceptions in the case narrative. The method blank and all related field and QC samples shall be re-extracted/re-analyzed. Acceptability may be determined by citing historical sample and method results on a case by case basis.

9.4 Summary of QC

- 9.4.1** Method Blank (Mblk)
9.4.1.1 Detected concentrations < PQL or <CDL
9.4.1.2 Detected concentrations < 10X amount in associated samples
- 9.4.2** LCS: (Minimum) Every 20 Samples
9.4.2.1 Recovery within lab historical limits
9.4.2.2 Recovery within lab historical limits
- 9.4.3** LCSD if insufficient sample volume for MS/MSD
9.4.3.1 Recovery within lab historical limits, %RSD <30%
- 9.4.4** MS/MSD:
9.4.4.1 Recovery within lab historical limits %RSD <30%, failures noted in job comments, no action required
- 9.4.5** Surrogates TCX & DCBP:
9.4.5.1 All Samples and Quality Control - One surrogate recovery within lab historical limits, none below 10%.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	Statistical Limits ⁴
Matrix Spike (MS) ²	1 in 20 or fewer samples	Statistical Limits ⁴
MS Duplicate (MSD) ²	1 in 20 or fewer samples	Statistical Limits ⁴
Surrogates	every sample ³	Statistical Limits ⁴

¹ LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

² The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

³ Analytical and QC samples (MB, LCS, MS/MSD)

⁴ Statistical control limits are updated annually and are updated into LIMS.

9.5 Instrument QC

- 9.5.1** ICAL:
9.5.1.1 608 - %RSD < 10% or linear R factor >0.995, (R² >0.990)
9.5.1.2 8081 - %RSD < 20% or linear R factor >0.995, (R² >0.990)
- 9.5.2** CCV: ICV (second source): Within +/-20% of true value

9.6 Corrective actions for Out-of Control Data

9.6.1 Indicate routine corrective actions if control limits not met for each of the following

9.6.1.1 ICAL Analysis can not begin without an acceptable calibration curve. Instrument maintenance may be required

9.6.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained

9.6.3 CCV: Reanalyze the CCV for external standard method.

9.6.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

9.6.3.2 If 2nd analysis is unacceptable, analyze a new ICAL or instrument maintenance may be needed.

9.6.3.3 If internal standard method is used, all results prior to the failing CCV are acceptable. (up to 20 injections)

9.6.4 Method Blank: Re-extract all samples associated with an unacceptable method blank.

9.6.5 LCS

9.6.5.1 If below limits: Re-extract all samples associated with an unacceptable LCS

9.6.5.2 If above limits: Re-extract all samples with detections of Pesticides. Re-extraction not required if samples are ND.

9.6.6 MS/MSD:

9.6.6.1 Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

Recoveries in both MS and MSD are consistent (%RSD<30)
and the LCS recovery is acceptable

If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation shall be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager shall be included in the final decision process prior to releasing data.

9.6.7 Surrogate:

9.6.7.1 All Samples and Quality Control - One surrogate recovery within lab historical limits, none below 10%, comment only.

9.6.7.2 Lab Quality control - Both surrogates out, rerun, still out, batch re-extraction

9.6.7.3 Samples – Both surrogates out without matrix effects or historical support, re-extraction required.

9.6.7.4 Samples – Leachate, Waste or Historically supported matrix effects, report and add job comment

ICAL: %RSD < 20% or calibration factor >0.995

ICV (second source): Within +/-20% of true value

CCV: %D < 20%

If the CCV is out high and there are no positives in the samples the results may be reported. It must, however, be noted in the logbook and on the Job Summary.

Method Blank:

Detected concentrations < PQL

Detected concentrations < 10X amount in associated samples

LCS:

Recovery within lab derived historical limits.

*DEP RPC limits must be within 40-140%

MS/MSD:

Recovery within lab derived historical limits.

Surrogate:

Recovery within lab historical limits.

*DEP RPC limits must be within 30-150%

9.7 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

9.7.1 Acceptability may be determined by citing historical, sample, and method results on a case by case basis.

9.7.2 In the event acceptable data can not be obtained, a Non-Conformance Memo (NCM) must be filed with the Project Manager for notification of the client.

10 Procedure

10.1 Sample Preparation

10.1.1 Extraction Refer to SOPs BF-OP-004 (Continuous Liquid-Liquid Extraction), BF-OP_005 (Aqueous Separatory Funnel Extraction Procedure), BF-OP-003 (Method 3550-Ultrasonic Extraction Procedure), and Project-specific extraction procedure for guidance in choosing the appropriate extraction procedure.

10.1.2 Spiked samples and Matrix Spike Blank(s) are used to verify the applicability of the chosen extraction technique to each extraction batch and to determine the accuracy of the procedure. For Pesticide analysis, the spike solution consists of all single component pesticides listed in Table 2 with the exception ADDS.

10.1.3 Gel Permeation Cleanup (SOP BF-OP-009) and Florisil Cartridge Cleanup (SOP BF-OP-007) for Pesticides are not routinely performed, unless specifically requested in a project specific quality assurance plan.

Step	Standards	Type	Control Limit	Frequency
Method # 608				
<i>Initial Cal</i>	<i>3 Point Ical Minimum</i>	<i>Calibration Curve</i>	<i>%RSD , <10% or Linear >0.990</i>	<i>When ICV criteria cannot be reached with routine maintenance</i>
<i>Second Source (ICV)</i>	<i>Single Point</i>	<i>Alternate Lot# or Vender</i>	<i><15% D</i>	<i>After each calibration</i>
<i>Degradation Check</i>	<i>Single Point</i>	<i>Performance Evaluation Mix</i>	<i><20% degradation of Endrin and <20% degradation 4,4'-DDT</i>	<i>Daily</i>
<i>CCV</i>	<i>Single Point</i>	<i>Midrange Standard</i>	<i><15% D</i>	<i>Prior to analysis of samples</i>
<i>CCV</i>	<i>Single Point</i>	<i>Midrange Standard</i>	<i><15% D</i>	<i>Every 10 injection for external standard. Prior to every 20 injections for Internal Standard.</i>
Method # 8081A/B				
<i>Initial Cal</i>	<i>5 Point Ical Minimum</i>	<i>Calibration Curve</i>	<i>%RSD <20% or Linear >0.990</i>	<i>When ICV criteria cannot be reached with routine maintenance</i>
<i>Second Source (ICV)</i>	<i>Single Point</i>	<i>Alternate Lot# or Vender</i>	<i><20% D (8081B) <15% D (8081A)</i>	<i>After each calibration</i>
<i>Degradation Check</i>	<i>Single Point</i>	<i>Performance Evaluation Mix</i>	<i><20% degradation of Endrin and <20% degradation 4,4'-DDT</i>	<i>Daily</i>
<i>CCV</i>	<i>Single Point</i>	<i>Midrange Standard</i>	<i><20% D (8081B) <15% D (8081A)</i>	<i>Prior to analysis of samples</i>
<i>CCV</i>	<i>Single Point</i>	<i>Midrange Standard</i>	<i><20% D (8081B) <15% D (8081A)</i>	<i>Every 10 injection for external standard. Prior to every 20 injections for Internal Standard.</i>

10.2 Calibration

10.2.1 A five-point calibration curve is established for each compound of interest and must be reanalyzed whenever calibration verification cannot be achieved. The 5 levels for all single component pesticides are 0.005/0.010/0.050/0.100/0.15 ng/ μ l. These values are generated by using the Method Detection Limit (MDL) Study for the instrument, the working range of the detector, and the concentration required to meet routine detection limits based on the low standard in the calibration curve. This gives an expected low final concentration of 0.05ug/L for all water analysis given a sample volume of 1 Liter, and 1.667 ug/Kg for all soil analysis given a sample weight of 30.0g and 100% dry.

10.2.1.2 Calculate the calibration factor (CF) for each compound at each concentration level.

$$CF = \text{Peak Area} / \text{Standard Concentration (ng)}$$

10.2.1.3 Calculate the percent Relative Standard Deviation (%RSD) between the calibration factors of each compound at all concentration levels.

$$\%RSD = [CF \text{ Standard Deviation} / CF \text{ Mean}] \times 100$$

10.2.1.4 If the %RSD for each analyte is <20% the curve (608 <10%)can be assumed to be linear and the average Calibration Factor can be used to calculate the concentration of any compounds detected during sample analysis

10.2.1.5 If the %RSD exceeds 20% for any compound, concentrations of that compound found in samples must be calculated using linear regression. The correlation coefficient of this linear fit must be greater than 0.995.

10.2.1.6 The ICIS will be designated to the midpoint of the calibration (using the IS recovery for this run to link to all other CCVIS standards for reference)

10.2.2 The concentration of the standard used for calibration verification is prepared at the midpoint of the five point initial curve. (Below the midrange)

10.2.2.1 Single Component Analysis is performed after every 10 samples and at the end of a particular sequence of samples. Multi-component analytes will be run weekly when they are target analytes to insure pattern recognition. When identified, they will be verified either before or after the sample containing the positive in as timely a manner as possible.

10.2.2.2 The calibration factors of the components in the standard hall all be less than or equal to $\pm 20\%$ Difference (D) of the average response factor of the initial calibration curve.

$$\%D = [\text{Avg. } CF_{(\text{Initial})} - CF_{(\text{Cal.Verif.})} / \text{Avg. } CF_{(\text{Initial})}] \times 100$$

10.2.2.3 When using linear regression rather than %RSD, the %Difference values are calculated based upon nanogram values rather than calibration factors.

10.2.2.4 Failure to meet the average 20% D criteria for calibration verification may require recalibration of the instrument (new initial curve) and re-analysis of all samples analyzed since the last compliant calibration verification. If the CCV exhibits an elevated response, and the samples show no positives, the data may be used to show non-detects in the samples affected.

10.2.2.5 Cases where the CCV exhibits a %D > 20% but < 30%, can be utilized when known matrix affects consistently produces these results, either presently or historically. The exceptions will be noted in reference to the specific compounds and samples that are affected.

10.3 Retention Time Windows

10.3.1 The width of the retention time window for each compound is determined based upon the standard deviation of three retention time measurements of the individual standards measured over the course of a 72-hour period.

10.3.1.1 Make three injections of all single component standards mixtures and multi-component products throughout a 72-hour period.

10.3.1.2 Make Calculate the standard deviation of the three absolute retention times of each single component compound. For multi-component compounds, choose one major peak and calculate the standard deviation of the three absolute retention times for that peak. If the standard deviation calculates to be negligible or impractical, a default retention time of +/-0.05minutes is to be used for all single component pesticides, +/-0.07 minutes for all multi-components, and +/- 0.10 minutes for DCBP surrogate.

10.3.1.3 The retention time window is defined as ± 3 times the Standard Deviation of the absolute retention time for each standard.

10.3.2 Retention time windows are useful tools in compound identification, however experience of the analyst should weigh heavily in the interpretation of the chromatograms. For multi-component standards, the analyst should use the retention time window but should primarily rely on pattern recognition.

10.3.3 Retention time windows are to be calculated for each standard on each GC column on an annual basis or whenever the chromatographic conditions change, (ie: a new GC column is installed or the instrument is subjected to major maintenance).

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	$\leq 20\%$ RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 20 Injections ISTD (10 injections ESTD)	$\leq 20\%$ RSD 8081B $\leq 15\%$ RSD 608/8081A
RT Windows (RTW)	Init. CCV determines midpt. of RTW	$\pm 3X$ SD

10.4 **Sample/ Gas Chromatographic Analysis**

10.4.1 Prime the instrument, if needed, with two injections of 0.15 ng/μl pesticides mix standard, or hexane blank.

10.4.2 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. The breakdown of DDT and Endrin is measured before a pesticide curve or samples are analyzed for pesticides, and then every 12-hours or twice daily while running. Injector maintenance and/or recalibration is performed if the breakdown is greater than 20% for either compound.

10.4.2.4 Calculate percent breakdown as follows:

% breakdown for DDT = $\frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{peak areas (DDT + DDE + DDD)}} \times 100$

% breakdown for Endrin = $\frac{\text{Total Endrin degradation peak area (Endrin Aldehyde + Endrin Ketone)}}{\text{peak areas (Endrin + Aldehyde + Ketone)}} \times 100$

10.4.3 A 0.05 ng/μl standard of single component pesticide mix standard solution is analyzed every 10 samples. The response of the standard shall be within 15 % of the initial calibration.

10.4.4 The routine daily sequence shall consist of (at minimum):

PEM (Degradation Check)
8081 std (Midpoint Calibration Verification)
10 Samples (including QC)
8081 std (Midpoint Calibration Verification)
10 Samples (including QC)
8081 std (Midpoint Calibration Verification)

The multicomponent Toxaphene and Chlordane are run weekly at 0.50ng/ul (MidPoint) to verify the patterns. Adds (Table 4) compounds are injected as needed for quantitation.

10.4.5 Depending on the nature of the sample extracts and the target compounds required by the client, standards can be run more often to confirm any suspected response changes, and to verify potential positives. By using prescreening techniques, the sample extracts can be run diluted and/or interspersed with hexane blanks to allow continuous operation.

10.5 **TestAmerica Buffalo Dual Column Approach**

10.5.1 Buffalo adheres to the requirements stated in TestAmerica Corporate Policy CA-T-P-003: 'Reporting Results for Methods That Require Second Column Confirmation'. Since there are several options presented in this policy, the option and specific details used by Buffalo are presented here.

- 10.5.2** The laboratory routinely designates a primary column based on optimal separation of compounds of interest and other desirable characteristics (in which case the 2nd column is the confirmation column for each analyte). Both columns are intended to be calibrated and meet verification requirements.
- 10.5.3** If the difference between the dual column results is $\leq \pm 40\%$ RPD, report results from the primary column.
- 10.5.4** If there is a surrogate recovery or CCV problem for one of the two samples and not for the other report all results for that sample from the column with the acceptable QC.
- 10.5.4.4** If the CCV recovery for the primary column is within acceptance limits and the analyte is confirmed on the secondary column, but the second column is outside of acceptance limits, then the primary column may be reported with a case narrative comment or footnote.
- 10.5.4.5** If the CCV recovery for the confirmation column is within acceptance limits, and the recovery for the primary column is in the range of $+15$ to $+40\%$, then the results may be reported from the confirmation column with a case narrative comment or footnote.
- 10.5.4.6** If the CCV recovery is below acceptance limits for the primary column, or the secondary column is below acceptance limits and the analyte is not confirmed, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed with acceptable CCVs.
- 10.5.4.7** If the CCV recovery on either column is $>40\%$ above acceptance limits, any associated sample positives will need to be reanalyzed with acceptable CCVs.
- 10.5.5** The sample and QC values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.
- 10.5.6** If the calculated values are $>40\%$ RPD from each other, the data must be evaluated more closely to determine which value should be reported and an appropriate comment must be noted in the job summary and report case narrative.
- 10.5.6.4** Where surrogates are similar on both columns but there is a large difference ($>40\%$) between the sample values, the difference may be due to a positive interference on the column with the higher result. In this case the lower of the two numbers should be reported.
- 10.5.7** If the surrogates are very different between columns ($>40\%$ RPD), this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstance in the job summary and report case narrative.
- 10.5.8** If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.5.8.4 An exception to this requirement would be if the CCV recovery on one column were elevated and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

11.0 Calculations / Data Reduction

The concentration of specific analytes in samples is determined using the Average Calibration Factor (ACF) calculated from the initial curve.

AQUEOUS CALCULATION

CF = Peak Area of Standard / Standard Concentration (ng)
(Determine the calibration factor for each compound in the initial curve and then calculate the ACF)

NG of Compound in Sample = Peak Area of Compound / ACF

SOIL/SOLID CALCULATION

CF = Peak Area of Standard / Standard Concentration (ng)
(Determine the calibration factor for each compound in the initial curve and then calculate the ACF)

NG of Compound in Sample = Peak Area of Compound / ACF

UG/KG of Comp. = $\{[NG \times DF \times FV \times GPC] / [ExW \times DW]\} \times 1000$

UG/L of Compound = $\{[NG \times DF \times FV] / [ExV]\} \times 1000$

DF = Dilution Factor

FV = Final Volume of Concentrated Extract (mls)

ExV = Extraction Volume (mls)

DF = Dilution Factor

FV = Final Volume of Concentrated Extract (mls)

GPC= A factor of 2 is incorporated if GPC is used

ExW= Extraction Weight (grams)

DW = Decimal Dry Weight

Nanograms values may also be determined based upon the linear regression of the initial curve.

Calibrations, surrogates, spikes, method blanks, and chromatograms are reviewed by the analyst on a 100% basis to determine acceptability of the data. Through Turbochrom, the signals are downloaded into the TALs system, where the final calculation is performed electronically.

11.1 Accuracy

$$11.1.1 \text{ ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$11.1.2 \text{ MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.1.3 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

$$11.1.4 \text{ Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance**12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19.7 of the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

A one time Demonstration of Capability is executed to document the ability to perform the method. The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

13.0 Pollution Control

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

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. The following waste streams are produced when this method is carried out.

The following waste streams are produced when this method is carried out.

- Aqueous waste generated in the lab.
- Solvent waste generated by the extraction
- Vials containing sample extracts.

All solvent waste generated by the extraction is to be disposed of in a labeled "C" waste container.

All acidified aqueous waste is to be disposed of into a labeled "A" waste container.

15.0 References / Cross-References

15.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; SW846, Third Edition, Final Update III; Method 8081A; December 1996.

16.0 Method Modifications: NA

17.0 Attachments

17.1 Table 1: Standard Concentrations – Single Component

17.2 Table 2: Standard Concentrations – Multi-Component

17.3 Table 3: Standard Concentrations - Surrogates

17.4 Table 4: Target Compound List

17.5 Table 5: Column Elution & Relative RT Order

18.0 Revision History

Revision 3, August 31, 2015

- o Added IS references for reagents and standards
- o Added ISTD options and requirements to CCV sections
- o Combined 8081A CCV criteria into appropriate sections
- o In Section 9.3.1 removed reference to USACE.
- o Laboratory Director Change, signature added

Revision 2, September 29, 2014

- o Section 10.5 - Updated dual column reporting policy
- o Added Table for Elution order and relative RTs
- o Section 9.2.2.1 - added 608 MS frequency requirement

Revision 1, February 11, 2013

- o Quality Manager Change, signature updated.
- o Section 7.1.5.2 Added LVI-250ml extraction reference
- o Section 16.0 – Cited above in method modifications.

TABLE 1

STANDARD CONCENTRATIONS – SINGLE COMPONENT

<i>Standard Level</i>	<i>Concentration (ng/ul)</i>	<i>Conc. in sample - Water (1L)</i>	<i>Conc. in sample - Soils (30g/100% dry)</i>
A	0.005	0.05 ug/L	1.667 ug/kg
B	0.01	0.10 ug/L	3.334 ug/kg
C*	0.05*	0.50 ug/L	16.67 ug/kg
D	0.10	1.0 ug/L	33.34 ug/kg
E	0.15	1.5 ug/L	50.01 ug/kg

*** Mid Point Concentration is used for CCVs and Spikes -0.05ng/ul** (Mid range of Calibration curve is 0.075ng/ul)

TABLE 2

STANDARD CONCENTRATIONS – MULTI-COMPONENT

<i>Standard Level</i>	<i>Concentration (ng/ul)</i>	<i>Conc. in sample - Water (1L)</i>	<i>Conc. in sample - Soils (30g/100% dry)</i>
A	0.05	0.50 ug/L	16.67 ug/kg
B	0.10	1.0 ug/L	33.34 ug/kg
C*	0.50*	5.0 ug/L	166.7 ug/kg
D	1.0	10.0 ug/L	333.4 ug/kg
E	1.5	15.0 ug/L	500.1 ug/kg

*** Mid Point Concentration is used for CCVs and Spikes -0.50ng/ul** (Mid range of Calibration curve is 0.75ng/ul)

TABLE 3

STANDARD CONCENTRATIONS - SURROGATES

<i>Standard Level</i>	<i>Concentration (ng/ul)</i>	<i>Conc. in sample - Water (1L)</i>	<i>Conc. in sample - Soils (30g/100% dry)</i>
<i>A</i>	<i>0.005</i>	<i>0.05 ug/L</i>	<i>1.667 ug/kg</i>
<i>B</i>	<i>0.01</i>	<i>0.10 ug/L</i>	<i>3.334 ug/kg</i>
<i>C</i>	<i>0.05</i>	<i>0.50 ug/L</i>	<i>16.67 ug/kg</i>
<i>D</i>	<i>0.10</i>	<i>1.0 ug/L</i>	<i>33.34 ug/kg</i>
<i>E</i>	<i>0.15</i>	<i>15 ug/L</i>	<i>50.00 ug/kg</i>

TABLE 4

Target Compound List

Single Component Pesticides

Aldrin
 α -BHC
 β -BHC
 δ -BHC
 γ -BHC
 γ -Chlordane
 α -Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Endrin Ketone
Methoxychlor

ADD Compounds

Hexachlorobenzene
Alachlor
Chlorobenzilate
Mirex
2,4'-DDD
2,4'-DDE
2,4'-DDT

Multi Component Pesticides

Chlordane (Tech Chlordane)
Toxaphene

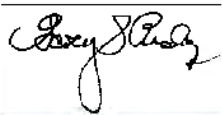
Column Elution & Relative RT Order

Column			
RTX-CLPI		RTX-CLPII	
rt	compound	rt	compound
2.25	Tetrachloro-m-xylene	2.47	Tetrachloro-m-xylene
2.64	alpha-BHC	2.96	alpha-BHC
2.87	gamma-BHC (Lindane)	3.26	gamma-BHC (Lindane)
2.94	beta-BHC	3.33	beta-BHC
3.08	delta-BHC	3.67	delta-BHC
3.26	Heptachlor	3.69	Heptachlor
3.52	Aldrin	4.01	Aldrin
4.09	Heptachlor epoxide	4.59	Heptachlor epoxide
4.21	gamma-Chlordane	4.79	gamma-Chlordane
4.34	alpha-Chlordane	4.94	alpha-Chlordane
4.44	4,4'-DDE	4.99	Endosulfan I
4.45	Endosulfan I	5.12	4,4'-DDE
4.72	Dieldrin	5.28	Dieldrin
4.96	Endrin	5.58	Endrin
5.05	4,4'-DDD	5.71	4,4'-DDD
5.18	Endosulfan II	5.80	Endosulfan II
5.33	4,4'-DDT	6.03	4,4'-DDT
5.62	Endrin aldehyde	6.14	Endrin aldehyde
5.85	Methoxychlor	6.42	Endosulfan sulfate
6.06	Endosulfan sulfate	6.72	Methoxychlor
6.34	Endrin ketone	6.95	Endrin ketone
7.37	DCB Decachlorobiphenyl	8.20	DCB Decachlorobiphenyl
2.50	Hexachlorobenzene	2.81	Hexachlorobenzene
3.64	Alachlor	3.84	Alachlor
4.08	2,4'-DDE	4.73	2,4'-DDE
4.60	2,4'-DDD	5.26	2,4'-DDD
4.86	2,4'-DDT	5.53	Chlorobenzilate
5.06	Chlorobenzilate	5.58	2,4'-DDT
5.94	Mirex	6.87	Mirex

Title: Analysis of PCBs
SW846 8082A/ 8082 / 40CFR 608

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Approvals (Signature/Date):



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1.0 Scope and Application

This method is used to quantify polychlorinated biphenyls (PCBs) in extracts from aqueous, soil, sludge or oil matrices by direct injection techniques into a capillary column equipped gas chromatograph. An electron capture detector (ECD) is employed for identification and quantification. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the integration of gas chromatograms.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is used to determine volatile organic compounds in a variety of matrices: water, soil, sediment, sludge and waste drum samples.

Compound	CAS No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The routine reporting limits are:	Sample	FV	Method
0.5 ug/L for water samples	1 Liter	10 ml	8082
	250ml (LVI)	2ml	8082
0.06 ug/L for low level water samples	1 Liter	2 ml	608/8082LL
1ppM for routine soil samples	2.0 grams	ml	8082
2.5 mg/Kg for routine oil/waste samples	0.2 grams	ml	8082
1 ug/wipe for wipe sample	1 Wipe	ml	8082

2.0 Summary of Method

2.1 Water & Wastewater samples: approximately 1 liter of sample is extracted with Methylene chloride using a separatory funnel (SOP BF-OP-003 3510) or a continuous liquid-liquid extractor (SOP BF-OP-004 3520). Another option is to utilize a 250ml initial volume and reduce to a final volume of 2ml to attain the same RL as a routine 1 Liter extraction

2.1.1 Soil samples are extracted using approximately 2.0g of soil/solid sample using 3550 sonication (SOP BF-OP-016) or 3546 Microwave (SOP # BF-OP-018)The extract is then exchanged to hexane and concentrated to 10 ml or less. The final extract is then separated by gas chromatography and detected by an electron capture detector. (specific low level requested RLs can utilize 30g of soil)

2.2 Florisil & Silica Gel column cleanup procedures and sulfur removal procedures may be utilized to mitigate any interferences that may be encountered during analysis. Although these procedures may eliminate several interferences, contamination of the sample may

come from a variety of sources, including solvents, reagents, glassware and any of the hardware used in sample processing. For this reason, reagent and solvent blanks should be analyzed to ensure their purity.

3.0 Definitions

3.1 Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual.

3.1.1 PCB (Polychlorinated Biphenyl)

The general term used to describe a mixture of congeners, generated via the manufacturing process.

3.1.2 Aroclor

Another reference to the PCB type, given as a known mixture rather than group of congeners.

3.1.3 Ar-

An abbreviated version of Aroclor, used as Ar1242 (Aroclor 1242).

3.1.4 Congener

Any of the specific individual "Parts" of a PCB, designated by the number of chlorines and the various isomers of each.

4.0 Interferences

4.1 Method interferences can be minimized by proper glassware cleaning methods, instrument maintenance, and the use of high purity reagents and solvents.

4.2 Sulfuric acid (SOP BF-OP-010 3665) is part of the extraction procedure for all PCB samples.

4.3 Copper cleanup Method 3660 (AGE-BF-GE-005), Silica Gel Cleanup (SOP BF-OP-008 3630), and Florisil Cartridge Cleanup (SOP BF-OP-007 3620) may be also used on samples when specified by project or historical results warrant further cleanup.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.4.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to near room temperature prior to working on them.

5.4.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.5 Primary Materials Used

5.5.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the safety Data Sheets (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Sulfuric Acid	Carcinogen Irritant Dehydrator Poison Oxidizer	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Safety Data Sheets (SDS) maintained in the laboratory

Aroclors have been classified as a potential carcinogen. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure.

6.0 Equipment and Supplies

Gas chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.

Capillary columns

ZB-35 30m 0.53mm w/0.5um film or Equivalent (if separation criteria is met)

ZB-5 30m 0.53mm w/1.0um film or Equivalent (if separation criteria is met)

ZB-35 30m 0.32mm w/0.5um film or Equivalent

ZB-5 30m 0.32mm w/1.0um film or Equivalent

Electron Capture Detector

PE Nelson Totalchrom data system (Version 6.2.1 or Later)

Carrier Gas Hydrogen

Make Up Gas - Argon/Methane or Nitrogen

Syringes – 10ul Teflon tipped for Injection

Chrom chromatography analysis system

6.1 Instrumentation *

Hewlett Packard 5890 gas chromatograph w/dual ECD detectors

Hewlett Packard 6890 gas chromatograph w/dual uECD detectors

Hewlett Packard 7673 Auto Samplers

Hewlett Packard 7683B Auto Samplers

Hewlett Packard 3396A Integrators

PE Nelson Totalchrom 6.2.1 data system

PE Nelson 900 Series A/D Boxes

PE Nelson 600 Series Link Controller Boxes

(* or Equivalent)

6.2 Supplies

Carrier Gas Hydrogen

Make Up Gas - Argon/Methane or Nitrogen

Syringes – various

1.8 Crimp-top Vials, Amber & Clear

5 ¾" Disposable Pipettes& Bulbs

Vila Inserts - 250ul Spring

Inlet Liners - Packed Purge w/Pesticide Grade Glass Wool

Inlet Liners – Capillary Drilled Uniler

2-4 um Extract Filters

7.0 Reagents and Standards

- 7.1** Reagents or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

- 7.2 Standards are stored in the GC Standard Incubator at $4\pm 2^{\circ}\text{C}$ in Teflon-sealed amber containers in the dark.
- 7.3 All stock standard solutions are replaced before the expiration date. All other standard dilutions or working standards are discarded after six month (or at the stock standard expiration date, whichever comes first) or sooner if routine QC indicates a problem. Certified PCB Mixes (Aroclors 1016/1260, 1221, 1232, 1242, 1248, 1254, 1262 & 1268)
- 7.4 Second Source PCB Mixes for all Aroclors (Different Manu. or Lot #) to verify constant response of newly prepared calibration curve or single point standards.
- 7.5 ISTD 1-bromo-2-nitrobenzene
- 7.6 Acetone (pesticide grade)
- 7.7 Hexane (pesticide grade)

8.0 Sample Collection, Preservation, Shipment and Storage

- 8.1 Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.
- 8.2 Aqueous Samples (608 & 8082) are to be collected in a 1-liter amber glass jar and stored at $4\pm 2^{\circ}\text{C}$. Organic preparation is to be performed within 1 year of collection.
- 8.3 Soil (solid) samples are to be collected in a 4 oz jar and stored at $4\pm 2^{\circ}\text{C}$. Organic preparation is to be performed within 1 year of collection.
- 8.4 Wipe and Oil samples are to be collected in a 4 oz jar and stored at $4\pm 2^{\circ}\text{C}$. Organic preparation is to be performed within 1 year of collection.
- 8.5 Analysis of all extracts is to be performed within 40 days of preparation.
- 8.6 Holding times specified in project specific quality assurance plans may supersede the above listed method criteria.
- 8.7 Extracts are stored in an Incubator under refrigeration at $4\pm 2^{\circ}\text{C}$.

9.0 Quality Control

- 9.1 Sample QC - The following quality control samples are prepared with each batch of samples.
 - 9.1.1 A method blank must be prepared and analyzed with each batch (maximum 20 samples). The acceptance criterion is that the method blank must contain a

concentration less than the reporting limit for all target analytes. If the blank fails this criterion, the entire prep batch must be re-extracted and analyzed.

- 9.1.2** Analysis of at least one matrix spike and one matrix spike duplicate per batch. Sample spike and duplicate recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data. If the recoveries are not achieved, the data is still valid as long as the matrix spike blank is acceptable. The routine matrix spiking solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (LCS/MS/MSD).
- 9.1.3** Limited sample volume can allow for the analysis of a matrix spike blank duplicate instead of a MS and MSD pair.
- 9.1.4** A matrix spike blank must be prepared and analyzed with each batch. Spike recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data.
- 9.1.5** If Internal Standard ISTD is utilized, the ISTD of 1-bromo-2-nitrobenzene is added to all standards, quality control, and samples prior to analysis at a concentration of 0.5ng/ul.

9.2 Instrument QC

9.2.1 Initial Calibration Curve and Verification (ICC & ICV)

9.2.1.1 An Initial Calibration Curve (ICC) must be run for the Aroclor mix 1016/1260. Other Aroclors specified by project may be required if utilizing non-linear calibration models.

- 9.2.2** The curve will consist of a minimum of five concentration points ranging from 0.025ng/uL–2.0ng/uL. The concentration points will be prepared by diluting a certified Aroclor standard. Lower Levels may be prepared at the time of calibration by diluting existing higher levels. The following table summarizes the concentration levels used and the associated reporting levels for water, soil, wipe, & oil samples. If Internal Standard is utilized, 0.5ng/ul of 1-bromo-2-nitrobenzene is added to all standards.

9.2.2.1 An Initial Calibration Curve must be run for the surrogate compounds Decachlorobiphenyl and Tetrachloro-meta-xylene, which is to be contained in the Ar1016/1260 calibration standards, or can be analyzed separately, but must be contained in all Ar1660 calibration standards.

9.2.2.2 The ICC for the surrogates will require a minimum of five (5) concentration points. The concentrations will be made by serial dilutions of a certified standard. These levels are included within the Ar1660 calibration standards, recommended levels are: 0.00125ng, 0.0025ng, 0.005ng, 0.0125ng, 0.025ng, and 0.05ng.

9.2.2.3 A single point calibration must be run for all other Aroclors. The Aroclor concentration should be near the midrange concentration of the 1016/1260 curve (~0.5ng/ul on column).

9.2.2.4 If utilizing a non-linear calibration for Ar1660, multiple point calibrations for all quantified Aroclors must also be utilized.

9.2.3 Continuing Calibration Verification (CCV)

9.2.3.1 If Internal Standard methodology is employed, the initial CCV can be followed by up to 20 Environmental Samples or 12 hours, whichever comes first, and does not require a closing CCV for compliance. A Continuing Calibration Verifications (CCV) must bracket every 10 samples for ESDT methodology. Aroclor 1660 is used to represent all Aroclors for this purpose.

9.2.3.2 A CCV for each identified Aroclor should be analyzed daily or within 12 hours of the sample. If criteria is met, no reanalysis is necessary for any positives.

9.2.4 Instrument Blank (IBLK)

9.2.4.1 An Instrument Blank (IBLK) is analyzed immediately after the Continuing Calibration Verification (CCV) to verify the absence of any potential carry-over or contamination.

9.2.5 Calibration Acceptance Summary

9.2.5.1 Retention Time Windows

9.2.5.1.1 The Initial RT Window Study is calculated using 3X the Standard Deviation (in minutes) of 3 injections of the Ar1660 Midpoint Standard (0.5ng) over a 72-hour period.

9.2.5.1.2 With the use of EPC (Electronic Pressure Control), the values given from this study will most likely be impractically small or 0.00. Any peak with a 3X Standard deviation of <0.05ng minutes defaults to 0.05 min (3.0 sec) at minimum. It should be noted that the primary means of identification are based upon pattern recognition.

* Matrix effects may shift actual retention times, but the primary pattern may remain intact.

9.2.5.1.3 The calibration retention time windows may be adjusted slightly (tighter or wider) to better reflect peak width values, to allow for consistent situational identification and quantification. When RT drift due to maintenance or minor changes conditions occurs, these windows should be centered and updated for each Aroclor simultaneously, based upon a full set of all Aroclor

standards, to allow these values for each component to reflect the current chromatographic conditions.

9.2.5.2 Calibration Model

- 9.2.5.2.1 For 8082A/8082, the Percent Relative Standard Deviation for Aroclor1016 and Aroclor1260 must be $\leq 20\%$ for the ICC to be acceptable, and to use single point standards for the remaining Aroclors. ($\leq 10\%$ for 608).

For 608, a calibration of 3 points is required, for 8082A/8082, 5 points, and If a quadratic regression fit is required to obtain acceptable data, 6 or more calibration points must be employed. If a 2nd Order Curve Calibration for Ar1660 is used, single points for the remaining Aroclors cannot be used to calculate sample positives. If Aroclor positives are found, then the calibration requirements for full curves are then applied to any such Aroclor.

- 9.2.5.2.2 If $RSD \leq 20\%$ ($\leq 10\%$ for 608).for each Aroclor (1016 & 1260), then linearity of the detector can be assumed for all other Aroclors over the same analytical range. The congener range for Aroclor 1016 reflects the ranges for Aroclors 1221, 1232, 1242, & 1248. The congener range for Aroclor 1260 reflects the ranges for Aroclors 1254, 1262, & 1268.
- 9.2.5.2.3 The RSD for some of the individual peaks of each Aroclor 1016 & 1260 can be $>20\%$ and $<30\%$, as long as the Total Aroclor RSD for each is $<20\%$. ($\leq 20\%$ total $\leq 10\%$.for 608).
- 9.2.5.2.4 For each Aroclor, five major chromatographic peaks are chosen (except Ar1221 which requires three peaks) that represent the key to the patterns present in each particular Aroclor for quantification. Five peaks will be used in calibration to allow for the potential loss of peaks due to interferences. This is important due to the assessment of degraded patterns when determining identification and quantification needs in difficult matrices.
- 9.2.5.2.5 *Is has been found that "Unique" Peaks for each Aroclor do not exist, due to the fact that many mixes contain all the congeners for another Aroclor.*
- 9.2.5.2.6 **Due to these facts, the following combinations of Aroclors cannot be identified simultaneously in a given sample:**
- 9.2.5.2.7 Aroclors: 1016, 1232, & 1242 will not have unique peaks that meet all criteria when compared to one another.
- 9.2.5.2.8 Aroclors: 1221 & 1232 will not have unique peaks that meet all criteria when compared to one another.

- 9.2.5.2.9 Aroclors: 1260 & 1262 will not have unique peaks that meet all criteria when compared to one another.
- 9.2.5.2.10 The remaining Aroclors; 1248, 1254, & 1268 can be identified in combination, and along with any one of the other of the single Aroclors listed above.
- 9.2.5.2.11 It is important to note that the identification of multiple Aroclors in any given sample can be difficult and requires a vast working knowledge of the distinct parts of each pattern. It is paramount that the majority of the biphenyls present are to be explained using the most representative pattern match, along with the best quantitation of peaks present. The outside factors such as weathering, dechlorination, matrix, and overlap must also be considered when identifying potential Aroclors in complex sample patterns.
- 9.2.5.2.12 The Percent Relative Standard Deviation for each surrogate must be $\leq 20\%$ ($\leq 10\%$ for 608), or have a Correlation Coefficient "R" ≥ 0.995 (R squared ≥ 0.990) for the ICC to be acceptable. The curve may be determined linear if a minimum of 5 points are used, and quadratic if a minimum of 6 points are used.
- 9.2.5.2.13 The resolution of the triplet in the latter pattern of Aroclor 1260 is to be $\leq 75\%$ on one of the two columns used.

10.0 Procedure

- 10.1** Set up the Hewlett Packard Gas Chromatograph as a single injection split into dual column/detector analysis for each instrument.
 - 10.1.1** Split injection instruments shall have different columns as to maximize the ability to confirm Aroclors present in the extracts in the most efficient manner.
 - 10.1.2** Acceptable ICC's and single point calibrations are run for all Aroclors and surrogates. Both sides of a split injection instrument must be calibrated with the identical injections.
 - 10.1.3** The Packard Gas Chromatograph will require priming prior to use if allowed to sit idle for more than 24 hours.
 - 10.1.3.1 Priming consists of analyzing several recently injected standards and/or hexane blanks to allow the oven and other high temperature zones to equilibrate.
 - 10.1.4** An ICV will consist of a concentration point at or near the midrange of the curve, (generally 0.5ng on column). The concentration point will be prepared through serial dilutions of a certified second source Aroclor standard.

- 10.1.4.1 The response factor of the ICV must be $\pm 20\%$ D for Aroclors 1016 & 1260 for method 8082A ($\pm 15\%$ for 8082/608)
- 10.1.4.2 The CCVs may be biased high to confirm non-detects. ICVs and CCVs that are biased low will require a sample and system evaluation to determine if the effects are temporary or lasting.
- 10.1.4.3 In the temporary case, the instrument should be baked out and allowed to come to equilibrium to check compliance.
- 10.1.4.4 If the symptoms still persist, routine maintenance of liner, septa, syringe, rinse vial, guard column or other replacement may be required prior to checking compliance.
- 10.1.4.5 If the system still in non-compliant, check the instrument for stability by analyzing several test runs, prior to analyzing a new calibration curve. Detector Maintenance, changing the Column, or changing the detector settings will require recalibration of the system.
- 10.1.4.6 Sample analysis which continually produces reduced response upon re-analysis can be dealt with in several ways
 - 10.1.4.6.1 The extracts should be checked for possible further cleanup and/or dilution to minimize these effects.
 - 10.1.4.6.2 The samples may be diluted for color, due to the presence of metallic or non-carbon compounds which do not respond to electron capture detector, and have severely adverse effects upon the equipment.
 - 10.1.4.6.3 The insertion of hexane blanks after some samples may allow the instrument to recover. This step should not be done as routine without historical or screening data to support it, otherwise all analysis within bracketing CCVs need to be analyzed in a similar manner. Where Internal Standard methodology is employed, blanks may be interspersed at the analyst's discretion.
- 10.1.4.7 Sample analysis which continually produces reduced response, historically/site based, or have known matrix effects which are unaffected by any cleanup methodology may lead to:
 - 10.1.4.7.1 Dilutions due to these facts, not based upon matrix, positives, color, or any other visual data.
 - 10.1.4.7.2 Review of Report Level requirements and submittal of data as analyzed, with comment that the data is to be considered bias low
 - 10.1.4.7.3 Client notification, if unaware of these potential effects upon the data from this project or site. Decisions then going forward should always consider client feedback for future analysis and actions.
- 10.1.5** The retention time window determined for each peak must be adjusted using the CCV on a daily basis to account for any shifts in the instrument's operating conditions.
- 10.1.5.1 The 1016/1260 mix contains all of the unique components (or congeners) of each individual Aroclor and if the CCV is acceptable for this, it can be understood that the CCV is acceptable for all Aroclors.

10.1.6 Continuing Calibration Verifications (CCVs) for Ar1016/1260 must bracket every 10 samples. Other Aroclors may be analyzed along with this ICV/CCV midpoint standard to confirm full pattern recognition. These additional standards are not subject to quantifiable verification. Where Internal Standard methodology is employed, only a CCV prior to every 20 Environmental Samples or 12 hours is required.

10.1.6.1 For 8082A, the response factor for each Aroclor in the CCV must be $\pm 20\%$ D. ($\pm 15\%$ for 608/8082) All data is acceptable as long as it is bracketed by an acceptable ICV and CCV, or CCV and CCV. Where Internal Standard methodology is employed, only a CCV prior to every 20 Environmental Samples or 12 hours is required.

10.1.7 Each sample is injected into the gas chromatograph and its acceptable data is evaluated for Aroclor patterns and surrogate recovery. Spike recovery is also evaluated in spiked samples.

10.2 Sample Preparation

10.2.1 The most commonly used extraction procedures are SW-846 Methods 3510 Sepfunnel (waters SOP # BF-OP-003) , 3550 Sonication (soils SOP # BF-OP-005), and 3546 Microwave (soils SOP # BF-OP-018),

10.2.2 Prior to sample analysis, the extracts should be screened on a GC with an ECD setup, to better judge any potential dilutions, cleanups, or high level contamination dangers. This one-time analysis will be based loosely upon the response seen from a midrange standard analyzed in the same manner.

10.3 Sample Analysis

10.3.1 MB: A laboratory method blank must be analyzed with every set of 20 samples at a minimum of 1 per batch. Acceptance criteria are less than the report limit. If the acceptance criteria are met, the QC sample indicates no contamination due to the preparation procedure and is considered acceptable. If analyte is measured above the reporting limit, reanalyze. If reanalysis is acceptable, continue. If reanalysis again indicates contamination the sample results are not useable for drinking water samples. Results for other sample matrices may be used if they are greater than 10 times the blank contamination.

10.3.2 Blank contamination and recoveries outside this range may lead to: Re-extraction if within holding time and volume available, noting recoveries in case narrative or flagging values as estimated. The spike results, sample matrix, and reported positives in the prep batch are also to be considered. The Project Manager will be notified with a job exception, and acceptability may be determined by citing historical, sample, and method results on a case by case basis.

10.3.3 LCS: A laboratory control sample (lab fortified blank) must be analyzed with every batch of 20 samples or a minimum of 1 per day. The routine matrix spiking

solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (LCS/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract.

- 10.3.4** Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the required recovery limits are met, the QC sample indicates control of the preparation procedure and is considered acceptable. If the recovery limits are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met the batch results are not useable unless the control sample recovery is high and the sample concentrations are below the reportable limit.
- 10.3.5** MS: A matrix spike sample must be set for one in every batch of 20 samples. Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the acceptance criteria are met, no adverse matrix effects are indicated. If acceptance criteria are not met, continue and this result will be noted in the case narrative in reference to a compliant Matrix Spike Blank. To minimize bias, samples for matrix spike analysis shall be chosen at random. All analytes in the spike solution shall be measured unless they are not of interest in the spiked sample.
- 10.3.6** MSD: Along with every matrix spike sample, a duplicate MS must also be set. This sample is the matrix spike duplicate (MSD). Acceptance criteria are <30% RPD. If the acceptance criteria are met, continue. If the acceptance criteria are not met, continue and this result will be noted in the case narrative in reference to a compliant Matrix Spike Blank.
- 10.3.7** When QC results, unknown positives, or sample matrix present the analyst with questionable data, the spike results, sample matrix, and reported positives in the prep batch are all to be considered. Acceptability may be determined by citing historical, sample, and method results on a case by case basis. The project manager shall be notified of any method anomalies, and can then contact the client as to specific instructions on the usability of the data and any further actions.
- 10.3.8** Sample analysis is primarily based upon a single primary column. The results from the column chosen are deemed to be the best data chromatographically, and yield the most consistent values. The PCB analysis primarily is focused upon correct identification from the patterns to match Aroclors. From these pattern matches, a determination can then be accomplished to verify the presence above a reporting limit for any given Aroclor. This is then confirmed on the secondary column, via a positive result above the MDL, as long as the confirmatory column is not out of compliance in a decreased value. Confirmatory patterns are key to assessing a true PCB pattern match, therefore this is the primary focus of the dual column approach. Matrix effects, Degradation, Multiple Aroclor presence, and Non-Target compounds can complicate the quantitation to where the actual unfettered number of any single Aroclor can only at best be estimated. It is also best practice not to remove high level peaks from a summarized total, given the fact that it is not known whether some peaks are

lower due to degradation, elevated due to dechlorination, overlap, or a combination of the three. It is the conservative approach to identify as many potential Aroclors present, even given to bias high results, to best serve the environmental decisions made from the results.

10.4 Example Analysis Queue ISTD Method

ID	Comments
Priming Runs	Startup
All Individual Aroclors	CCV(1660) other Aroclors for RT update only
IBLK/Hexane	Instrument Blank
20 Environmental Samples (IS method)	10 injections for ESTD method
Ar1660 & IBLK	Closing CCV only required for ESTD
20 Environmental Samples (IS method)	10 injections for ESTD method
Ar1660 & IBLK	Closing CCV only required for ESTD
20 Environmental Samples (IS method)	10 injections for ESTD method
Ar1660 & IBLK	Closing CCV only required for ESTD

11.0 Calculations / Data Reduction

11.1 Include all formulas used to calculate/interpret data. Other documents may be referenced. The QA Manual may contain many of the more frequently used formulas. Include any guidance to be used when interpreting the data. You may include examples in the Attachment section.

11.2 Examples:

Accuracy

$$\text{CCV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

$$\text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

Calculating the ng amount of Aroclor in each peak.

$$\frac{\text{Area of the peak (a)}}{\text{Calibration factor of peak (a)}} = \text{ng amount of peak (a)}$$

(or the ng amount from the curve equation if linear calibration)

Calculating the ng amount of Aroclor in the sample

$$\frac{\text{Peak(n)ng} + \text{peak(n+1)ng} + \text{peak(n+2)ng} + \dots}{\text{Total Peaks}} = \text{ng amount of sample}$$

Converting ng amount to ug/Kg, ug/L and ug/wipe

$$\text{ug/Kg} = \frac{(\text{ng}) \times (\text{final volume in ml}) \times (\text{dilution factor})}{1000} \times \frac{1}{(\text{injection vol. in ul}) \times (\text{sample wt.}) \times (\% \text{ Dry})}$$

For Oils % dry = 100

$$\text{ug/L} = \frac{(\text{ng}) \times (\text{final volume in ml}) \times (\text{dilution factor})}{(\text{injection volume in ul}) \times (\text{sample volume in L})}$$

$$\text{ug/wipe} = \frac{(\text{ng}) \times (\text{final volume in ml}) \times (\text{dilution factor})}{(\text{injection vol. in ul}) \times (\text{sample wt.}) \times (\% \text{ Dry})}$$

For wipes, sample weight = 1, % Dry = 100

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 MDL studies are performed annually on a matrix and instrument type basis

12.1.2 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.1.3 MDL studies are to be performed using Aroclor 1016/1260 as the representative mixture for each applicable matrix, and then for the range of biphenyls found in all of the remaining reported Aroclors. The MDLs generated for Aroclor 1016 will be reflective for the ranges for Aroclors 1221, 1232, 1242, & 1248. The MDLs generated for Aroclor 1260 will be reflective for the ranges for Aroclors 1254, 1262, & 1268.

12.1.4 MDLVs (Verifications) will be analyzed for all Aroclors, for every matrix, on all Instruments, to verify the recovery and allowable reporting of the applied MDLs above to each representative Aroclor. These should be spiked at no greater than the RL equivalent concentration on-column, and should show a positive ng value on the analysis report for all Aroclors.

12.2 Demonstration of Capabilities

12.2.1 An initial demonstration of capability (IDOC) is performed for either aqueous or soil matrices per analyst and compared to the method criteria. The concentration used is either equal to a CCV or a LCS.

12.2.2 The analyst will run, analyze, and report 4 standards or spikes. Reporting the expected concentrations of each in a summary report. This is usually entered into a LIMS data system for reporting, and a final copy is submitted to the QA department for record keeping

12.2.3 A continuing demonstration of capability (DOC) will be performed on an annual basis for each analyst, for each operational method they run and analyze.

12.3 Training Requirements

12.3.1 The QA Manual or a Training SOP may be referenced for training requirements. If applicable, state required concentration of samples prepared for Precision and Accuracy study or alternate training procedure.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability)

13.2 Waste streams produced by this method

13.2.1 Acidic waste generated in the lab.

13.2.2 Solvent waste generated by the extraction

13.2.3 Expired primary and working PCB standards

13.2.4 Vials containing sample extracts

13.2.5 Solid Wastes

14.0 Waste Management

14.1 If the published method does not include this section, a statement similar to the following may be inserted:

14.2 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.3 All solvent waste generated by the extraction is to be disposed of in a labeled "C" waste container.

14.4 All acidified aqueous waste is to be disposed of into a labeled "A" waste container

14.5 All Solid Wastes are to be disposed of in to the labeled "BE" waste containers.

15.0 References/Cross- References

15.1 Methods," 3rd edition, SW-846, update III, Dec. 1996. Method 8082A U.S. Environmental Protection Agency, Office of Solid Waste and Energy Response, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd edition, SW-846, update IV, February 2007.

16.0 Method Modifications:

- Designation of a Primary Column for quantification, and the secondary column for confirmation only.

17.0 Attachments None

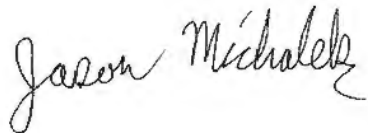
18.0 Revision History:

- Revision 2, dated July 30, 2015
 - o Updated Lab Director, signature added
 - o Integrated ISTD options into method
 - o Added Microwave extraction option
 - o Added TestAmerica Minimum Standard requirements
 - o Specified 8082A/8082/608 individual requirements
- Revision 1, dated February 11, 2013
 - o Updated QA Manager
 - o Revised section 1.1 to include 1ppm Waste extraction.
 - o LVI references added
 - o Enhancement of dual column assessment, confirmatory data, and quantification.
- Revision 0, dated March 31, 2008
 - o Integration for TestAmerica and STL operations.

**Title: Analytical Methods for GC/MS Semivolatile Samples by SW846
8270D**

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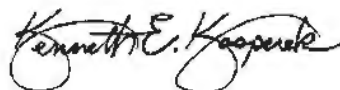
Approvals (Signature/Date):



3/11/15

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1.0 Scope and Application

This SOP contains the procedures for the determination of extractable semi-volatile organic compounds (SVOC) by gas chromatography/mass spectrometry (GC/MS).

The routine matrices performed by this procedure are waters and soils. Other matrices which may be performed include wipes, leachates, tissues, and wastes.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

2.0 Summary of Method

A measured volume or weight of sample is extracted using separatory funnels or sonication extraction procedures. The extract is then analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses characteristic as compared to component reference spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis of the target compounds is performed by the internal standard technique using a single characteristic

3.0 Definitions

Additional definitions can be found in the TAL Buffalo Laboratory Quality Manual (LQM)

4.0 Interferences

4.1 Some of the possible interferences that arise during GCMS Semivolatile analysis include, but are not limited to:

1. Glassware contamination
2. Matrix interference
3. Aldol condensation
4. System air leaks
5. Injection port/liner contamination
6. Warped filament, and/or dirty source and rods
7. APIX analytes Methapyrilene and Phentermine split at all concentrations and require manual integration in calibration standard.

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the Laboratory SOP to ensure it is free from contaminants and does not contribute artifacts.

High purity reagents and solvents are used to help minimize interference problems. Acetone and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Chemicals that have been classified as carcinogens or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

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

Analysts are expected to use caution and common sense while working in a laboratory environment. Each employee is required to read the companies' Corporate Safety Manual. All of the samples to be analyzed have the potential to contain hazardous substances. Most standards also contain hazardous chemicals and many do contain known carcinogens. Employees must use protective equipment when handling standards, samples and extracts including gloves, lab coats and safety glasses. It is the analyst's responsibility to read and familiarize themselves with the MSDS of each chemical and/or reagent involved in this method.

Samples, standards and/or extracts should never be opened or transferred outside of a fume hood.

Waste disposal is all C waste with the exception of some acids used in the cleaning of equipment which is disposed of in AN waste.

Spills should be cleaned up promptly and waste should be disposed of as per the Chemical Hygiene Plan.

There is also the danger of burns while doing repair or maintenance on a gas chromatograph. One must use caution while working on or near the injection port or transfer line.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Calibrated micro syringes 10, 25, 50, 100, 500, 1,000 microliter.

6.2 2ml amber vials and caps.

6.3 Disposable pipets and pipet bulbs.

6.4 Volumetric flasks.

6.5 Instrumentation

Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.5.1 Gas Chromatograph -

- Hewlett Packard 6890
- Carrier gas Helium UPC grade or equivalent

6.5.2 Gas Chromatography Column

- Analysis: Phenomenex ZB-Semivolatiles GUARDIAN (cat# 7HG-G027-11GCC) or equivalent

6.5.3 Mass Spectrometer

- HP5973 and HP5973 inert
- Tuning compound PFTBA
- Scan Range 35-500 AMU/second

6.5.4 Data System

- HP Chemstation
- Chrom chromatography analysis system
- TALS data analysis system

7.0 Reagents and Standards

7.1 Methylene Chloride – high purity

7.2 Standards:

7.2.1 Stock Standards

CLP Semivolatile Calibration Mix 1000µg/ml

Calibration Mix #2 2000µg/ml

Benzidines Mix 2000 µg/ml

N-Nitrosodiphenylamines 5000 µg/ml

OLM Mix 2000 µg/ml

Benzoic Acid 2000 µg/ml

BN/AP Surrogate Mix 4000 µg/ml

DFTPP mix 50µg/ml or equivalent (To be stored away from light and @ 6 degrees C when not in use)

Internal Standard Mix 2.0 mg/ml

All Certificates of Analysis received from the manufacturer are maintained in a laboratory LIMS system. Stock standards are prepared every twelve months or sooner, if necessary.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc.	Final Conc. In Standards
CLP Semi-Volatile Calibration Mix Calibration Mix # 4	MECL ₂	1000 ng/ul	400µl	2000ul	200 ng/ul	200 ug/L
Benzidines Mix	MECL ₂	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
N-Nitrosodiphenylamine Mix	MECL ₂	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
BN/AP Mix	MECL ₂	5000 ng/ul	80µl	2000ul	200 ng/ul	200 ug/L
OLM Mix	MECL ₂	4000 ng/ul	100µl	2000ul	200 ng/ul	200 ug/L
Benzoic Acid	MECL ₂	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
	MECL ₂	2000 ng/ul	400µl	2000ul	200 ng/ul	400 ug/L

7.2.2 Initial and Continuing Calibration Solutions 8270 Stock Solution

7.2.3 Working Standards

7.2.3.1 Surrogate Standard Spiking Solution is prepared that contains nitrobenzene-d5, terphenyl1-d14, 2-fluorobiphenyl, and 1,2-dichlorobenzene-d4 at a concentration of 100µg/ml; phenol-d5, 2,4,6-tribromophenol, 2-fluorophenol and 2-chlorophenol-d4 at a concentration of 150µg/ml. Surrogate standards are added to all samples and calibration solutions. Additional surrogates may be added at the laboratory's discretion.

Standard	<i>Solvent</i>	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. In Standards
Semi-volatile Acid Surrogate Phenol-d5 2,4,6-Tribromophenol 2-Fluorophenol 2-Chlorophenol-d4	MEOH	10,000ng/ul	1,500ul	100,000ul	150ug/L
Semivolatile B/N Surrogate Nitrobenzene-d5 Terphenyl-d14 2-Fluorobiphenyl 1,2-Dichlorobenzene-d4	MEOH	5000ng/ul	2,000ul	100,000ul	100ug/L

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

- 8.1 Water samples may be collected in 1L or 250 mL amber glass containers with Teflon lined, screw-caps.
- 8.2 Soil/Sediment Samples may be collected in glass containers fitted with Teflon-lined screw-caps or closed end tubes.
- 8.3 All samples are stored at 4 C (+/-2C) from the time of collection until extraction.
- 8.4 Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.
- 8.5 Soil samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.
- 8.6 Sample extracts are stored at 4 C (+/-2C) in the SVOA sample extract refrigerator prior to analysis

9.0 Quality Control

- 9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	Statistical Limits ⁴
Matrix Spike (MS) ²	1 in 20 or fewer samples	Statistical Limits ⁴
MS Duplicate (MSD) ²	1 in 20 or fewer samples	Statistical Limits ⁴
Surrogates	every sample ³	Statistical Limits ⁴

¹ LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

² The sample selection for MS/MSD is randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

³ Analytical and QC samples (MB, LCS, MS/MSD)

⁴ Statistical control limits are updated annually and are updated into LIMS.

Method Blanks - A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate/clean sand for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

- 9.1.1 For semivolatile analysis, a method blank for water samples consists of 1 L volume of reagent water spiked with 1.0mL of the surrogate spiking solution. For the 8270 LVI method (Large Volume Injection) 250 mL of reagent water is spiked with 1 mL of the LVI surrogate solution and concentrated to a final volume of 1 mL. For medium or low level soil/sediment samples, a method blank consists of 1g or 30g

of sodium sulfate/clean sand spiked with 1.0mL of the surrogate spiking solution, respectively. Extract, concentrate, cleanup and analyze the blank according to procedures for water and soil samples.

9.1.2 Acceptance Criteria – levels of target analytes in the method blank must be less than the required reporting limit or less than one-tenth the concentration of the respective analyte in the associated samples. The MRL is set at either the MDL or the MDL Check.

9.1.3 Corrective Actions for Method Blank Analyses - If the acceptance criteria for method blank analysis are not met, the analytical system may be assumed to be out of control. The following corrective actions may be taken:

- If contamination exceeds the acceptance criteria listed in Section 9.1.2i, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvent, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the GC/MS be eliminated. Samples associated with the contaminated blank must be re-extracted and re-analyzed.
- If surrogate recoveries in the method blank do not meet the acceptance criteria, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the blank and all associated samples OR the samples may be reported as estimated, and noted in the case narrative.
- If the method blank does not meet internal standard response requirements, check calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the method blank. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the blank

9.2 Laboratory Control Sample/Matrix Spike/Matrix Spike Duplicate(LCS/MS/MSD)

9.2.1 A Laboratory Control Sample (LCS), matrix spike and matrix spike duplicate are analyzed to evaluate the analytical system and the effects of sample matrix on the methods used for semivolatile analysis.

9.2.2 The LCS, matrix spike, and matrix spike duplicate are spiked with the compounds of interest (at concentrations noted in the standard preparation section).

9.2.3 A LCS, matrix spike and matrix spike duplicate are extracted and analyzed for every batch of 20 samples of a similar matrix. Matrix spike and matrix spike duplicates are not performed for field QC samples such as rinsates, or field/trip blanks. Corrective action guidance for failures in the LCS are in section 9.2.8 of this SOP.

9.2.4 If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, duplicate Laboratory Control Samples may be processed.

9.2.5 Dilutions

Dilutions of MS/MSD samples are performed only if the unspiked sample requires a dilution in order to maintain any target compound concentrations in the upper half of the calibration. MS/MSD samples will not be diluted to get spiked or non-spiked compounds below the highest calibration standard. Any sample diluted 20x or greater will be deemed to have a low recovery and shall be qualified accordingly.

9.2.6 Calculations for MS/MSD

The concentrations of spiked compounds are determined using equations described for sample analysis. After determining the compound concentrations, the percent recovery is calculated using Equation 1.

Equation 1

$$\text{Matrix Spike Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

Where,

SSR= Spike Sample Result

SR = Sample Result

SA = Spike Added

The relative percent difference between the matrix spike and matrix spike duplicate is calculated using Equation 2.

Equation 2

$$\text{RPD} = \frac{[\text{MSR} - \text{MSDR}]}{1/2 (\text{MSR} + \text{MSDR})} \times 100$$

Where,

RPD = Relative Percent Difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference; hence RPD is always expressed as a positive value

9.2.7 Technical Acceptance Criteria for MS/MSD

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for matrix spike and matrix spike duplicate analysis also.

The matrix spike recovery limits are based on historical data and are updated annually.

The matrix spike recovery limits are advisory. If the recovery limits are not met, no further corrective action will be necessary. However, frequent occurrences of this nature should be investigated.

Re-extraction and re-analysis of the matrix spike and matrix spike duplicate may be necessary if, in the technical judgment of the analyst and/or supervisors, an error was made during the extraction procedure

9.2.8 Technical Acceptance Criteria for LCS:

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for the LCS analysis also. Any failures in the LCS are flagged automatically in the laboratories TALS LIMS system.

The Laboratory Control Sample recovery limits are based on historical data and are updated annually.

If the Laboratory Control Sample was found to be unacceptable it must be reanalyzed. If the re-analysis confirms the failing results then the impacted samples must be re-extracted and re-analyzed. In instances where the failures indicate a high bias in the LCS and the associated samples do not contain the affected analytes above the Reporting Limit the data is considered acceptable and maybe reported. If the sample was not within extraction hold time, a job exception must be filed and both analyses must be included with the report. However there are allowable failures based on the number of total compounds spiked. Any failures are flagged automatically by the laboratories TALS LIMS system.

Number of analytes in LCS	Number of marginal exceedances allowed
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

9.2.9 Surrogate Recoveries

The surrogate compound concentrations are determined using calculations found in Section 9.1.1. The recoveries are then determined using Equation 3

Equation 3

$$\% \text{ Recovery} = \frac{\text{Concentration (}\checkmark\text{ amount) found}}{\text{Concentration (}\checkmark\text{ amount) spiked}}$$

Recovery limits for surrogate compounds are based on historical data and are updated annually.

9.3

Instrument QC

Typical Instrument Operating Conditions are presented below. These may be modified as necessary to accommodate large volume injection (LVI) techniques which may utilize up to a 5 uL injection.

I

OVEN

Initial temp: 45 °C (On) Maximum temp: 340 °C
Initial time: 3.00 min Equilibration time: 0.20 min
Ramps:
Rate Final temp Final time
1 30.00 280 0.00
2 9.00 325 1.00
3 0.0(Off)
Post temp: 70 °C
Post time: 0.00 min
Run time: 16.83 min

FRONT INLET (SPLIT/SPLITLESS)

Mode: Pulsed Splitless
Initial temp: 250 °C (On)
Pressure: 15.42 psi (On)
Pulse pressure: 30.0 psi
Pulse time: 0.55 min
Purge flow: 50.0 mL/min
Purge time: 0.50 min
Total flow: 54.3 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

COLUMN 1

Capillary Column
Model Number: Phenomenex ZB-Semivolatile GUARDIAN (cat#7HG-G027-11)
Max temperature: 330 °C
Nominal length: 30.0 m (with integral 10m guard column)
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Mode: constant flow
Initial flow: 1.4 mL/min
Nominal init pressure: 15.43 psi
Average velocity: 37 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

FRONT DETECTOR (NO DET)

SIGNAL 1	SIGNAL 2
Data rate: 20 Hz	Data rate: 20 Hz
Type: test plot	Type: test plot
Save Data: Off	Save Data: Off
Zero: 0.0 (Off)	Zero: 0.0 (Off)
Range: 0	Range: 0
Fast Peaks: Off	Fast Peaks: Off
Attenuation: 0	Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

THERMAL AUX 2

Use: MSD Transfer Line Heater
Description: MSD Transfer Line
Initial temp: 325 °C (On)
Initial time: 0.00 min
Rate Final temp Final time
1 0.0(Off)

GC Injector

Front Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.00 - 4.00 microliters
Syringe Size	10.0 microliters
PreInj Solvent A Washes	0
PreInj Solvent B Washes	0
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

MS ACQUISITION PARAMETERS

General Information

Tune File : dftpp.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 2.60 min

EM Offset : 0
Resulting EM Voltage : 976.5

[Scan Parameters]

Low Mass : 35.0
High Mass : 500.0
Threshold : 100
Sample # : 2
A/D Samples 4

[MSZones]

MS Quad : 150 °C maximum 200 °C
MS Source : 230 °C maximum 250 °C °C

9.4 Instrument Performance Check

The GC/MS system is tuned using Perfluorotributylamine (PFTBA) such that an injection of 50ng of DFTPP will meet the abundance criteria listed in Table 2.

Prior to the analysis of standards or samples, the mass calibration and resolution of the GC/MS system is verified by the analysis of DFTPP. This analysis will verify the proper tuning of the system for 12 hours. After 12 hours, the instrument performance must be verified before standard and sample analysis may continue.

The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions.

Breakdown of 4,4'-DDT into 4,4'-DDD and 4,4'-DDE may be used to assess GC column performance and injection port inertness and must be less than 20%.

The compounds Benzidine and Pentachlorophenol should be present and at their normal responses for this concentration. Peak tailing should not be visible (PCP tailing factor <2 and Benzidine <2). If responses are poor and excessive peak tailing is present, corrective actions for the GC/MS instrument performance check solution may be required. Benzidine and Pentachlorophenol tailing may also be verified in the CCV.

All subsequent standards and samples must be acquired under the same GC/MS tuning conditions that were used for the analysis of the instrument performance check solution.

9.4.2 Technical Acceptance Criteria for the GC/MS Instrument Performance Check (DFTPP) is listed in Table 2.

9.4.3 Corrective Actions for the GC/MS Instrument Performance Check

If any of the acceptance criteria are not met, the DFTPP should be re-injected to insure that the injection made was not a cause for failure. If, after reinjection, acceptance criteria has not been met, one or more of the following corrective actions may be taken:

1. Retune the GC/MS
2. Clean the source; replace parts, etc...
3. Cut the column at the injector end
4. Replace the column
5. Replace the septum in the injector
6. Replace the injector liner
7. Clean injection port with MeCl_2
8. Change injection port seal
9. An instrument service call may be placed.

9.5 Initial Calibration

After the instrument performance check criteria has been met and prior to the analysis of samples, the GC/MS system is calibrated at a minimum of five concentration levels in order to establish instrument sensitivity and linearity.

The initial calibration shall be performed when major instrument maintenance has been performed or if continuing calibration criteria cannot be met.

Major instrument maintenance may consist of source cleaning, column changing, or quadrupole rod adjustment. Preventative maintenance such as septum changes, injector liner changes or column cutting may not require an initial calibration to be performed.

9.5.1 Procedure

Calibration standards for routine level 8270 (1 uL injection) are prepared to contain all target and surrogate compounds. A 20µl aliquot of the 2000 ug/mL internal standard solution is added to a 1mL aliquot of each calibration standard solution. The resulting concentration of internal standards is 40ng/uL. 8270 LVI (4 uL injection) calibration standards are spiked with 20uL of a 200 ug/mL internal standard solution resulting in a concentration of 4 ng/uL. The internal standards used are given in Table 3.

The relative response factors (RRF) for each target and surrogate compound is determined using equation 4. The characteristic ions for a given compound are listed in Tables 4 and 6. Internal standard assignments are listed in Table 3.

Equation 4

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured (see Table 3)

A_{is} = Area of the characteristic ion for specific internal standard (see Table 4)

C_{is} = Amount of the internal standard injected (ng)

C_x = Amount of the compound to be measured injected (ng)

The mean relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

Equation 5

$$\%RDS = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{(n-1)}}$$

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

9.5.2 Acceptance Criteria for Initial Calibration

If more than 10% of the compounds included in the initial calibration exceed 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, or the method specified compounds do not meet the minimum RRF acceptance requirements, then the system is deemed out of control and instrument maintenance should be performed to bring the instrument back into control. All minimum RRF requirements are listed as an attachment in the back of this SOP. The required 30% read back for the calibration is accomplished by the laboratories TALS LIMS system.

Any individual analyte result that fails the 20%/0.99 criteria must be flagged as estimated for any positive results.

9.5.3 Corrective Actions for Initial Calibration

If any of the acceptance criteria for initial calibration are not met, it may be necessary to reanalyze one or more of the calibration standards. If after reanalysis, the acceptance criteria have not been met, it may be necessary to take further corrective actions.

The following corrective actions may be taken if the acceptance criteria for initial calibration cannot be met.

1. Prepare fresh standards and reanalyze the initial calibration.
2. Replace the septum on the injector
3. Replace the injector liner
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Clean the source
7. An instrument service call may be placed

The acceptance criteria must be met before sample analysis may proceed.

9.5.4 Initial Calibration Verification

To verify the accuracy of the initial calibration, a standard is obtained from a source different from the calibration standards.

Immediately following analysis of an acceptable initial calibration curve, an aliquot of the second source standard with a concentration approximating the mid point of the curve of is injected

Recoveries of all compounds shall fall within the required 70-130% acceptance limit with the exception of the "Poor performers", whose criteria are listed as an attachment in the SOP.

9.5.5 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance is verified by the injection of a mid level standard.

The continuing calibration check must be analyzed once every 12-hour time period of operation. This check must be analyzed prior to the analysis of samples for a given 12-hour time period.

9.5.6 Procedure for Continuing Calibration

A mid level standard is used for the continuing calibration verification. The relative response factor is calculated using procedures described for initial calibration. In the attachment section of this SOP the list of required response factors is listed.

If quantitation is performed using response factor, calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target and surrogate compound using Equation 6.

Equation 6

$$\% \text{ Difference}_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

RRF_c = Relative response factor from continuing calibration standard

If quantitation is performed using a least squares regression or a non-linear model, calculate the concentration of all analytes and surrogates in the continuing calibration as described in section 8.3.2 of this SOP. Calculate the percent drift using Equation 7.

Equation 7:

$$\% \text{ Drift} = \frac{\text{Conc}_E - \text{Conc}_A}{\text{Conc}_E} \times 100$$

Where:

Conc_E = Expected Concentration

Conc_A = Actual Concentration

9.5.7 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for each must be greater than or equal to the response factors listed in Table 6 attachment labeled update 4 response factors found at the back of this SOP.

Internal Standard retention times and responses are evaluated after acquisition of the continuing calibration check. If the retention time of any internal standard shifts by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration or the response of any internal standard is outside of the -50% to +100% range compared to the mid-point standard level of the most recent initial calibration, the system shall be inspected and corrected as needed. The CCV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

9.5.8 Corrective Actions for Continuing Calibration

If any of the technical acceptance criteria for continuing calibration are not met, it may be necessary to reanalyze the continuing calibration standard. If after reanalysis the acceptance criteria cannot be met, further corrective actions may be required.

The following corrective actions may be taken if the acceptance criteria for continuing calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Replace injection port seal
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Prepare fresh standards
7. Reanalyze the initial calibration
8. If the retention time of the compounds shifts more than 30 seconds and all compounds can be identified and the continuing calibration is still acceptable, all analysis may proceed.

9.6.1 Continuing Calibration Acceptance Summary

For the continuing calibration verification to be valid; no more than 20% of the compounds can fail either the 20%D or %Drift criterion.

If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit

For any analyte that has a non-detect associated with a calibration that fails the 20% requirement/minimum response factor criteria there must be a demonstration of sensitivity at the reporting limit. The criterion for a passing demonstration of sensitivity is detection only, and the results less than the reporting limit may be reported without flagging, and results greater than the reporting limit must be flagged as an estimate.

10.0 Procedure

- 10.1 Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, continuing calibration and second source calibration verification requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

Internal standard solution is added to each sample extract. 20µL of internal standard solution is added to each accurately measured 1.0mL of water sample extract. For

soil/sediment samples and water samples subjected to GPC, 10µL of internal standard solution is added to each accurately measured 0.5mL of sample extract. This will result in a concentration of 40ng/µL of each internal standard. The amount of internal standard needs to be adjusted according to how much extract volume was present in the extract vial. The exact volume of extract is measured using a syringe. The amount of Internal Standard solution to be added is then adjusted accordingly. The calculation to determine the amount of IS to add is provided below:

$$\text{Vol. Extract (ml)} \times 20 \text{ ul} = \text{FV of IS}$$

Necessary dilutions are made prior to adding internal standard solution. The internal standard solution must be added so that the concentration of each internal standard is 40ng/µL.

10.2. Dilutions

Dilutions of sample extracts are required if any target compound exceeds the initial calibration range.

The dilution chosen should keep the response of the largest target compound within the calibration range.

Dilutions above 10x will be deemed to have too low a surrogate recovery and shall be qualified accordingly.

10.2.1 Sample Log Book Entry

Prior to the start of the analysis samples are logged into a unique LIMS worklist which serves as an electronic runlog. This is accomplished with a barcode scanner which scans barcodes supplied directly from TALS with unique sample identification.

10.3. Qualitative Identification

10.3.1 Target Compounds

Target compound identification is done by comparing the sample mass spectrum to that of the standard. The following criteria must be satisfied in order to verify identifications.

Elution of the sample analyte within GC relative retention time unit window established from the 12-hour calibration standard.

Correspondence of the sample analyte and calibration standard component mass spectra.

To establish correspondence of the GC relative retention time (RRT), the sample component RRT must compare with ± 0.06 RRT units of the mid level calibration. If samples are analyzed within the same 12-hour period as the initial calibration, the 50ng standard is used to verify relative retention times.

To establish correspondence of the sample component mass spectra to that of the standard, the following criteria must be met:

- All ions present in the standard mass spectrum at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.
- The relative intensities of ions specified in the paragraph above must agree within ± 30.0 percent between the standard and sample spectrum. (Example: For an ion with an abundance of 50.0 percent in the standard spectrum, the corresponding sample ion abundance must be between 20.0 and 80.0 percent).
- Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J".

If a compound does not meet all of the above criteria, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, the compound will be identified

10.3.2 Non-Target Compounds

A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH mass spectral library is used to identify non-target compounds of greatest apparent concentration by a forward search of the library. The following compounds will not be identified by a library search routine:

- a. Internal standard compounds
- b. Surrogate compounds
- c. Volatile target compounds

Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-7-pentanone and 4-methyl-3-pentene-2-one) are searched and reported as part of the 30 tentatively identified compounds.

10.3.3 Guidelines for Making Tentative Identifications

Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ± 30 percent. Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

If, in the technical judgment of the mass spectral interpretation specialist, no tentative identification can be made the compound will be reported as unknown. Further identification may be possible, such as molecular weights or classifications (i.e., unknown hydrocarbon, unknown acid, etc.)

Pesticide target compounds may be tentatively identified by a library search.

10.4 Technical Acceptance Criteria For Sample Analysis

The samples must be analyzed on a GC/MS system meeting the DFTPP initial calibration, continuing calibration, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the acceptance criteria for GPC calibration.

The sample must be extracted and analyzed within the holding times.

The sample must have an associated method blank meeting the blank acceptance criteria.

All Matrix Spike Blank recoveries must fall within the laboratory derived limits. Recoveries above the upper control limit are acceptable as long as the corresponding analytes are not detected in the associated samples above the quantitation limit.

All surrogates should fall within the laboratory derived limits (Up to one BN and/or one AP surrogate may fall outside the control limit as long as the recovery is greater than 10%).

The relative retention time of each surrogate must be within ± 0.06 RRT units of its relative retention time in the continuing calibration standard.

The instrumental response (EICP area) for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

The retention time shift for each of the internal standards must be within ± 0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the 12-hour standard calibration range unless a more dilute aliquot of the sample extract is also analyzed.

10.5 Corrective Actions for Sample Analysis

The technical acceptance criteria must be met before data are reported. Contamination from laboratory sources requires re-extraction and reanalysis.

10.5.1 Surrogate Compounds

If the technical acceptance criteria for surrogate compound recoveries is not met, the following corrective actions are taken in the given order:

- a. Calculations, injection volumes, preparation volumes are checked to insure that an error was not made; if all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.
- b. The sample is re-injected to insure that an error during injection was not made. If after re-injection, surrogate recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.
- c. The sample is re-extracted. Exceptions: (1) in the case where the recoveries in a sample, MS/MSD agree (i.e., all samples exhibited recoveries outside of criteria limits) it will be noted in the Case narrative. (2) Insufficient sample remains for re-extraction. In this instance, the client will be contacted in order to determine the next procedure to follow. If this situation should arise, it will be documented in the Case narrative. (see form B: Re-extraction request form).
- d. After re-extraction, the sample is re-injected. If after re-analysis surrogate recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error. If, after re-analysis surrogate recoveries are not within criteria limits, a matrix effect may be assumed. If this should occur, both analyses may be reported. The instance will be documented in the Case Narrative.

10.5.2 Internal Standard Compounds

If the technical acceptance criteria for internal standard recoveries is not met, the following corrective actions are taken in the given order:

- a. Calculations, internal standard solution volumes and injected volumes are checked to insure that an error was not made. If all calculations and volumes were correct the analyst will proceed to the next step in the corrective action process.

- b. The sample is re-injected to insure that the instrument was working properly. If after re-analysis, the internal standard recoveries are within criteria limits, the second analysis will be reported only. If after re-analysis the internal standard recoveries are outside of criteria limits, both analyses will be reported and it may be assumed that a matrix effect was involved. If this instance should arise, it will be documented in the Case Narrative.

Exception: If internal standard recoveries of a sample, MS/MSD agree (i.e., recoveries are outside of criteria limits for all three samples, it may be assumed that a matrix effect is involved and no corrective action is necessary. The instance will be documented in the Case Narrative.

10.5.3 Relative Retention Times

If the technical acceptance criteria for the relative retention times of the internal standard compounds or surrogate compounds are not met, the following corrective actions are taken in the given order:

- a. Carrier gas, zone temperatures and instrument temperature programs are checked to insure that an error was not made or that the gas tank was not dry or clogged. If no errors are found the analyst will proceed to the next step in the corrective action process.
- b. The sample is re-analyzed to insure that an error was not made during the first injection. If, after reanalysis, the relative retention times are not within the technical acceptance criteria, it may be assumed that a matrix effect was involved. Both analyses will be reported and the instance will be documented in the Case Narrative. If, after re-analysis, the relative retention times are within the technical acceptance criteria, the second analysis will be reported only.

Exception: If the relative retention times of a sample, MS/MSD agree (i.e., relative retention times are outside of criteria limits for the sample, MS and MSD, it may be assumed that a matrix effect was involved and further corrective action is not necessary.

10.5.4 Laboratory Control Spike

If the Laboratory Control Spike was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. Marginal exceedance rules apply as listed in the SOP, section 9.2.8. Compounds listed in the poor performer attachment of this SOP do not count towards the marginal exceedances. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

10.6 Injection Logs: Injection Logs must contain the following information:

- Date, time, and analyst initials
- File number (FRN), sample ID, vial #, and work order #
- Injection volume, final volume, initial volume and dilution factor
- Indicate if tailing of degradation was present in the tune
- References for the standard, tune mix, IS mix
- Daily maintenance performed
- Any non-conformances with the samples

11.0 Calculations / Data Reduction

11.1 Target Compounds

Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (see Table 3). The EICP area of primary characteristic ions of analytes listed in Tables 4 and 6 are used for quantitation.

In instances where manual integration is necessary due to co-elution baseline noise or matrix interferences, all instances will be initialed and dated by the analyst. The quantitation report is documented as such by an "m" next to the compound that has been edited. In all instances of manual integration, a hardcopy of the EICP for that compound will be supplied with the raw data, this applies to all target compounds, internal standards and surrogate compounds.

The average response factor (RRF) from the initial calibration analysis (linear model) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, the reason is then documented in the case Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a relative factor is calculated using the secondary ion.

11.2 Water Samples

The following Equation (Eq. 8) is used to determine the concentration of target compounds identified in water samples:

Equation 8

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRFi)(V_o)(V_i)}$$

Where,

- A_x = Area of the characteristic ion for the compound to be measured
 A_{is} = Area of the characteristic ion for the internal standard
 I_s = Amount of internal standard injected in nanograms (ng)
 V_o = Volume of water extracted in milliliters (mL)
 V_i = Volume of extract injected in microliters (μL)
 V_c = Volume of the concentrated extract in microliters (μL) (V_c = 1,000 μL if sample was not subjected to GPC; V_i = 500 μL if sample was subjected to GPC)
RRFi= Relative response factor determined from the initial calibration
GPC= GPC factor.

GPC= 1.0 if water sample was not subjected to GPC;
 Df = Dilution factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0

11.3 Soil/Sediment Samples

The following Equation (Eq. 9) is used to determine the concentration of target compounds in soil/sediment samples:

Equation 9

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRF_i)(V_i)(W_s)(D)}$$

Where,

A_x , I_s , A_{is} are as given for water, above.

V_c = Volume of the concentrated extract in microliters (μL) ($V_t = 500 \mu\text{L}$)

V_i = Volume of the extract injected in microliters (μL)

$D = \frac{100 - \% \text{ moisture}}{100}$

W_s = Weight of sample extracted in grams (g)

GPC= GPC factor (GPC = 2.0 to account for GPC cleanup)

RRFi= Relative response factor determined from the initial calibration.

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for semivolatile by this method is defined as follows:

$$\frac{\mu\text{L most conc. Extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. Extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract not recovered from the use of GPC cleanup. Concentrating the extract collected after GPC to 0.5mL maintains the sensitivity of the soil/sediment method.

11.4 Tentatively Identified Compounds

Non-Target Compounds

An estimated concentration for non-target compounds tentatively identified is quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences is to be used. The equations for calculating concentrations are the same as equations 8 and 9. Total area counts (or peak heights) from the total ion chromatograms are used for both the compounds to be measured and the internal standard. A relative response factor (RRF) of one (1) is assumed. The resulting concentration is to be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (Presumptive evidence of presence), indicating the quantitative and qualitative uncertainties is calculated for all tentatively identified compounds as well as those identified as unknowns.

All TIC identification shall be entered in to the laboratory LIMS system, for QC blanks the “no TICS found” reportable box shall be shut off when reporting TICS due to the problems it causes on the analytical report.

11.5 Rounding is performed automatically in the LIMS system

11.6 Organic Significant Figures

For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is above 10.

12.0 Method Performance

12.1 Data assessment and acceptance criteria for quality control measures:

12.1.1 When internal standards are out of range a re-injection is required unless the problem can be determined to be a result of excessive matrix interference.

12.1.2 When surrogates are out of range, a re-extraction is required unless excessive visible chromatographic matrix interference is present. In this case, the Project Manager should be consulted to decide how to proceed.

12.1.3 When a positive hit for an analyte is above the calibration range a dilution must be performed to bring the value within calibration range.

12.1.4 When there are low spike recoveries in the matrix spike blank the entire extraction batch needs to be re-extracted. If there are high spike recoveries the associated sample data needs to be examined to assess if it may be biased.

12.2 Corrective actions for out of control data require Project Manager, Laboratory Director and/or QA Officer Notification. This can be accomplished either verbally, written using a Job Exception Report or both.

12.3 Contingency measures for handling out of control or unacceptable data requires the Project Manager to notify the client for input.

12.4 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the Corporate QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.5 Demonstration of Capabilities

Initial Demonstration of Capability (IDOC): The initial demonstration with each sample preparation and determinative method combination utilized must be performed by generating data of acceptable accuracy and precision for target analytes in a clean matrix. This is also done for new staff or when significant changes in instrumentation are made as stated in section 8.0 of Method 8000.

13.0 Pollution Control

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

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual. The following waste streams are produced when this method is carried out.

There are two types of aqueous waste generated in the lab:

1. A-Waste: All non-nitric acid and alkaline aqueous waste.
2. AN-Waste: All aqueous waste containing nitric acid.

These types of waste are to be disposed of into appropriately marked plastic containers.

The following are the other types of lab waste and where to dispose of:

1. C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards will also be dumped into C-waste cans.
2. Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.
3. Glass: contaminated glass needs to be rinsed off with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.
4. Extract Vials: extract vials are to be archived after they have been shot. After archival period, vials are to be crushed into a 55 gallon drum.

15.0 References / Cross-References

15.1 USEPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update IV, Method 8270D, 02/07.

16.0 Method Modifications: N/A

17.0 Attachments

17.1 Table 1: Semi-volatile Target Compound List and Contract Estimated Quantitation limits

17.2 Table 2: Ion Abundance

17.3 Table 3: Internal Standards and Corresponding Target Compounds Assigned for Quantitation

17.4 Table 4: Characteristic Ions for Target Compounds and Surrogates

17.5 Table 5: Poor Performing Compounds

17.6 Table 6: Update 4 Minimum Response Factors for selected compounds

17.7 Attachment A: SOP Procedure Summary

18.0 Revision History:

- Revision 2, Dated 11 March 2015
 - o Added LVI into sampling and preparation sections
 - o Added 8270 % drift requirements
 - o Updated instrument operation parameters
 - o Changed Department Manager, signature added
 - o Changed Lab Director, signature added
- Revision 1, Dated 11 March 2011
 - o QA Manager updated, signature added

17.1 TABLE 1 (Page 1 of 2)
Semivolatiles Target Compound List and Contract
Estimated Quantitation Limits

		Estimated Quantitation Limits		
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
34.	Phenol	108-95-2	5	170
35.	bis-(2-Chloroethyl)ether	111-44-4	5	170
36.	2-Chlorophenol	95-57-8	5	170
37.	1,3-Dichlorobenzene	541-73-1	5	170
38.	1,4-Dichlorobenzene	106-46-7	5	170
39.	1,2-Dichlorobenzene	95-50-1	5	170
40.	2-Methylphenol	95-48-7	5	170
41.	Bis(2-chloroisopropyl)ether	108-60-1	5	170
42.	4-Methylphenol	106-44-5	5	170
43.	N-Nitroso-di-n-propylamine	621-64-7	5	170
44.	Hexachloroethane	67-72-1	5	170
45.	Nitrobenzene	98-95-3	5	170
46.	Isophorone	78-59-1	5	170
47.	2-Nitrophenol	88-75-5	5	170
48.	2,4-Dimethylphenol	105-67-9	5	170
49.	bis(2-Chloroethoxy) methane	111-91-1	5	170
50.	2,4-Dichlorophenol	120-83-2	5	170
51.	1,2,4-Trichlorobenzene	120-82-1	5	170
52.	Naphthalene	91-20-3	5	170
53.	4-Chloroaniline	106-47-8	5	170
54.	Hexachlorobutadiene	87-68-3	5	170
55.	4-Chloro-3-methylphenol	59-50-7	5	170
56.	2-Methylnaphthalene	91-57-6	5	170
57.	Hexachlorocyclopenta-diene	77-47-4	5	170
58.	2,4,6-Trichlorophenol	88-06-2	5	170
59.	2,4,5-Trichlorophenol	95-95-4	10	330
60.	2-Chloronaphthalene	91-58-7	5	170
61.	2-Nitroaniline	88-74-4	10	330
62.	dimethylphthalate	131-11-3	5	170
63.	Acenaphthylene	208-96-8	5	170
64.	2,6-Dinitrotoluene	606-20-2	5	170
65.	3-Nitroaniline	99-09-2	10	330
66.	Acenaphthene	83-32-9	5	170
67.	2,4-Dinitrophenol	51-28-5	10	330
68.	4-Nitrophenol	100-02-7	10	330
69.	Dibenzofuran	132-64-9	5	170
70.	2,4-Dinitrotoluene	121-14-2	5	170
71.	Diethylphthalate	84-66-22	5	170
72.	4-Chlorophenyl-phenyl ether	7005-72-3	5	170
73.	Fluorene	86-73-7	5	170
74.	4-Nitroaniline	100-01-6	10	330
75.	4,6-Dinitro-2-methylphenol	534-52-1	10	330
76.	N-Nitroso-diphenylamine	86-30-6	5	170
77.	4-Bromophenyl-phenylether	101-55-3	5	170
78.	Hexachlorobenzene	118-74-1	5	170

		Estimated Quantitation Limits		
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
79.	Pentachlorophenol	87-86-5	10	330
80.	Phenanthrene	85-01-8	5	170
81.	Anthracene	120-12-7	5	170
82.	Benzyl Alcohol	100-51-6	5	170
83.	Di-n-butylphthalate	84-74-2	5	170
84.	Fluoranthene	206-44-0	5	170
85.	Pyrene	129-00-0	5	170
86.	Butylbenzylphthalate	85-68-7	5	170
87.	3,3-Dichlorobenzidine	91-94-1	5	170
88.	Benzo(a)anthracene	56-55-3	5	170
89.	Chrysene	218-01-9	5	170
90.	bis(2-Ethylhexyl)phthalate	117-81-7	5	170
91.	Di-n-octylphthalate	117-84-0	5	170
92.	Benzo(b)fluoranthene	205-99-2	5	170
93.	Benzo(k)fluoranthene	207-08-9	5	170
94.	Benzo(a)pyrene	50-32-8	5	170
95.	Indeno(1,2,3-cd)-pyrene	193-39-5	5	170
96.	Dibenzo(a,h)-anthracene	53-70-3	5	170
97.	Benzo(g,h,i)perylene	191-24-2	5	170
98.	Benzoic Acid	65-85-0	150	4800
99.	Benzaldehyde	100-52-7	5	170
100.	Acetophenone	98-86-2	5	170
101.	Caprolactam	105-60-2	5	170
102.	1,1'-Biphenyl	92-52-4	5	170
103.	1,2,4,5-Tetrachlorobenzene	95-94-3	5	170
104.	2,3,4,6-Tetrachlorophenol	58-90-2	5	170
105.	Atrazine	1912-24-9	5	170
106.	1,4-Dioxane	123-91-1	10	200
107.	N-Nitroso-di-n-butylamine	924-16-3	10	330
108.	N-Nitrosopyrrolidine	930-55-2	10	330
109.	o-toluidine	95-53-4	10	330
110.	Pyridine	110-86-1	25	330
111.	Dinoseb	88-85-7	20	330

17.2 TABLE 2

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10.0 – 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	0-100 percent of the mass 198
70	Less than 2.0 percent of mass 69
127	10.0 – 80.0 percent of mass 198
197	Less than 2.0 percent of mass 198
198	Base peak, or >50% of Mass 442
199	5.0-9.0 percent of mass 198
275	10.0-60.0 percent of mass 198
365	Greater than 1.0% of than mass 198
441	Present but less than <24% mass 443
442	Base Peak, or >50% of mass 198
443	15.0 – 24.0 percent of mass 442

17.3 TABLE 3
Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀	Phenanthrene-d	Chrysene-d ₁₂	Perylene-d ₁₂
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(b)fluoranthene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-nitroso-di-phenylamine	Butylbenzylphthalate	Benzo(k)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenylphenoether	3,3'-Dichlorobenzidine	Benzo(a)pyrene
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chlorophthalene	Hexachlorobenzene	Benzo(a)-anthracene	Indeno(1,2,3-cd)-pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	2-Nitroaniline	Pentachlorophenol	bis(2-ethyl-hexyl)phthalate	Benzo(g,h,i)-perylene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethylphthalate	Carbazole	Chrysene	Dibenzo(a,h)-anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene	Phenanthrene	Terphenyl-d ₁₄ (surr)	
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene	Di-n-octyl-phthalata	
4-Methylphenol	4-Chloroaniline	Acenaphthene	Di-n-butylphthalate		
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol	Atrazine		
2-Fluorophenol(surr)	2-Methylnaphthalene	Dibenzofuran			
Phenol-d ₅ (surr)	Nitrobenzene-d ₅ (surr)	2,4-Dinitrotoluene			
4-methylphenol	Benzoic acid	2,6-Dinitrotoluene			
Aniline	4-chloroaniline	Diethylphthalate			
Benzyl Alcohol	N-Nitrosobutylamine	4-Chlorophenyl-phenylether			
Benzaldehyde	Caprolactam	Fluorene			
Acetophenone	1,2,4,5-Tetrachlorobenz.	4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			
		1,1'-Biphenyl			
		2,3,4,6-Tetrachlorophenol			

17.4 Table 4 (page 1 of 3)
Characteristic Ions for Semivolatile
Target Compounds and Surrogates

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Benzyl Alcohol	108	79, 77
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Benzoic Acid	122	105, 77
SURROGATES		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

17.5 Table 5: Poor Performing Compounds

1,4 dioxane	N-nitrosodiphenylamine
Pyridine	Methane sulfonate
Benzaldehyde	1-naphthylamine
2-naphthylamine	N-nitrosodimethylamine
N-nitrosopiperidine	N-nitrosomorpholine
N-nitrosopyrrolidine	N-Nitrosomethylethylamine
p-phenylenediamine	p-Dimethylamino azobenzene
Methapyriline	a,a-dimethylphenethylamine
4-Chloroaniline	Aniline
3-nitroaniline	2-nitroaniline
2-Acetylaminofluorene	4-nitroaniline
3,3- dichlorobenzidine	3,3- dimethylbenzidine
2,4- dinitrophenol	Benzidine
Dinoseb	1,2-Diphenylhydrazine
Hexachlorocyclopentadiene	4-nitrophenol
Kepone	Hexachlorophene
Tetra Ethyl Lead	o,o,o-triethylphosphoro-thioate
4,6- dinitro-2-methylphenol	Phthalic Anhydride
Caprolactam	2,6 dinitrotolulene
Simazine	Famphur
4-nitroquinoline-1-oxide	Pentachlorophenol
(2)-9- octadecanamide	1,3,5 trinitrobenzene
1,3-Dinitrobenzene	Tri-cresylphosphate
2-Picoline	N,N Dimethylacetamide
Thionazin	Benzoic acid

*The laboratory's GC/MS semi-volatile's group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data as well as calibration data to validate each compound as a "poor performer". The criteria for corrective action with these compounds will be a less than 10% recovery for all compounds with the exception of Benzidine which will be less than 5%.

17.6 Table 6: Minimum response factors for selected compounds (Page 1 of 2)

Semivolatile compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6 Trichlorophenol	0.200
2,4,5 Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6 Dinitrotolulene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800

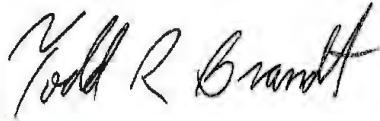
2,4-Dinitrotolulene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'- Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(g,h,i)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

17.7 ATTACHMENT A – SOP PROCEDURE SUMMARY

- I. Preparing the instrument;
 1. Cut column, change liner and septa, inject conditioning solution
 2. Ramp GC oven temp. to 325°C and ramp GC inj. Port pressure to 80 psi to see if pressure holds.
- II. Shoot DFTPP tune mix
 1. Shoot 1 ul of the dftpp tune mix
 2. Evaluate the DFTPP peak using the 4th Edition or criteria
 3. Evaluate the tailing factors of pentachlorophenol and benzidine.
 4. Evaluate the degradation of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE.
- III. Shoot single or 5pt. calibration;
 1. Shoot 1 ul of the 50ng continuing standard
 2. Evaluate the continuing; at least 80% of the compounds must have a %D less than 20 %
 3. If CCV does not pass criteria, then instrument maintenance must be performed, if this does not bring the system back in to control then a 5pt. curve (ICAL) must be shot.
- IV. Load Samples;
 1. Load blanks and LCSs in the beginning and dark samples toward the end.
 2. Very thick samples may be diluted.
 3. All samples must be shot within 12 hours of the tune injection.
- V. Analyze data;
 1. Quantitate all samples; need raw and enhanced spectra for positive and negative hits and 20 TICs .
 2. Shoot dilutions on any samples with positive hits over 160ng.
 3. Shoot reinjections (RI's) on any sample that has internal standards out, unless there is severe matrix interference that accounts for the low recovery.
 4. Samples with more than one BN or AP surrogate out needs to be re-extracted (RE).
- VI. CHROM/TALS Entry;
 1. Enter tunes, ICAL's and CCV's.
 2. Enter all samples to be included with the job.
 3. Identify and enter all TIC's
 4. Calculate, close and run data validator.
- VII. Review Data;
 1. Correct or explain any errors on the data validator.
 2. Make copies of logbooks, tunes, curves and standards and include them with the report.
 3. Check that all calculations have been made correctly.
 4. Turn in job for validation.

Title: Thermo Scientific ICAP 6500 Analysis
Method No(s). 6010C/CLP/200.7/6010B

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Approvals (Signature/Date):Todd Brandt
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1.0 Scope and Application

1.1 This SOP is specific for methods (SW-846) 6010C, 6010B, 200.7, and CLP and discusses the procedures as they are performed at TestAmerica Buffalo. It contains the procedures for the daily operation of the ICAP 6500 ICP-OES Spectrometer, and also contains procedures for calibration, standard, and sample preparation, instrument maintenance, data handling, and quality control.

1.2 At TestAmerica Buffalo, there are two ICAP 6500 ICP-OES Spectrometers equipped with ESI SC autosamplers. They are designated as ICAP1 and ICAP2. The ICAPs have both axial and radial viewing angles.

1.3 Analytes, Matrix(s), and Reporting Limits WELab

1.3.1 This SOP is used for the analysis of dissolved (soluble) water samples, digestates of total and dissolved waters, TCLP extracts, total recoverables, and digestates of soils, sludge, sediments, and other wastes.

1.3.2 The following elements are analyzed on each ICAP. Table 17.4 lists the wavelengths used for each ICAP.

Analyte Element	Symbol	Analyte Element	Symbol
Aluminum	Al	Manganese	Mn
Arsenic	As	Molybdenum	Mo
Antimony	Sb	Nickel	Ni
Barium	Ba	Sodium	Na
Beryllium	Be	Potassium	K
Boron	B	Selenium	Se
Cadmium	Cd	Silicon	Si
Calcium	Ca	Silver	Ag
Chromium	Cr	Strontium	Sr
Cobalt	Co	Thallium	Tl
Copper	Cu	Tin	Sn
Iron	Fe	Titanium	Ti
Lead	Pb	Vanadium	V
Lithium	Li	Zinc	Zn
Magnesium	Mg	Sulfur	S

1.3.3 Tables 17.2 and 17.3 list approximate Instrumental Detection Limits (IDLs) for each ICAP and achievable Method Detection Limits (MDLs). The laboratory IDLs are updated quarterly and the MDLs are updated annually, or when a significant change in instrumentation or methodology occurs. Current IDLs and MDLs are maintained in the laboratory LIMS.

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- 1.3.4 The laboratory standard Practical Quantitation Limits (PQLs) are also listed in Tables 17.2 and 17.3. The standard laboratory PQLs are only changed if there is a major update to the analytical system.
- 1.3.5 The linear range is the concentration range over which the instrument response to an analyte is linear. Table 17.5 lists the approximate linear ranges of each ICAP. Linear ranges are verified quarterly or when a significant change in instrumentation occurs.
- 1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

- 2.1 Samples and standards to be analyzed are digested or matrix matched to achieve an acidic aqueous solution containing 6% HNO₃ and 5% HCl by volume.
- 2.2 Samples are introduced to the instrument through an autosampler, combined with an internal standard, nebulized with argon gas to produce an aerosol, and transported to an argon plasma torch where sample excitation occurs. Characteristic atomic line emission spectra are produced by radio frequency inductively coupled plasma (ICP). Emission line intensity is measured by the instrument and processed by the instrument software (ITEVA). A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the emission line, are determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.
- 2.3 Normal steps in the daily operation of the ICAPs include:
- Perform any routine maintenance, if required.
 - Instrument start-up and warm-up, if instrument is not already conditioned
 - Preparation of standards (as needed). All calibration standards and quality control standards are prepared from stock solutions, with a 6 month expiration date.
 - Prepare all the samples for analysis including the required spikes, serial dilutions, and other quality control samples.
 - Set up an analysis run: a run is simply a sequence of samples to be analyzed with all required quality control samples, which are analyzed as a single unit.
 - Set-up the autosampler.
 - Analyze the samples.
 - When the analysis is complete, check the data for applicable method.
 - Enter analysis data into the LIMS.

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- Dispose of samples and standards appropriately. Clean-up lab area.

2.4 If the instrument is not operating properly or requires any maintenance, refer to Section 10.3 for help with routine maintenance and troubleshooting.

3.0 Definitions

3.1 ICAP – Inductively coupled argon plasma. Abbreviation used for Thermo Scientific ICAP 6500 ICP-OES Spectrometer.

3.2 IECs – Interfering Element Correction factors. Used to correct for interferences caused by spectral overlap of elemental lines. See Section 9.3.1 for procedures on determining IECs.

3.3 LDR - Linear Dynamic Range - also referred to as Linear Range (LR). The linear range is the concentration range over which the instrument response to an analyte is linear. Refer to Section 9.3.3 for the determination of linear ranges.

3.4 IDL - Instrument detection limit. The IDL of an element is the lowest calculated concentration that the instrument can measure. See section 9.3.2 for procedures on determining IDLs.

3.5 MDL – Method Detection Limit. The minimum concentration of an analyte that can be measured with a specified degree of confidence that the concentration is greater than zero.

3.6 PQL – Practical Quantitation Limit. The minimum concentration of an analyte that can be *quantitatively* measured with a specified degree of confidence and within *accuracy and precision guidelines*.

3.7 Calibration Standards - A series of solutions containing known amounts of each analyte within a matrix similar to samples. These solutions are used to calibrate the instrument.

3.8 ICV - Initial Calibration Verification – A standard used to verify the accuracy of the calibration, and which must be from a different source different from that of the calibration standard

3.9 ICVL – Low Level ICV, prepared at the same concentration as the Reporting limits for each analyte.

3.10 ICB - Initial calibration blank.

3.11 ICSA - Interference check sample containing only high levels of Al, Fe, Ca, and Mg.

3.12 ICSAB - Interference check sample containing high levels of Al, Fe, Ca, and Mg, and low levels of other elements that are analyzed by the ICAP.

3.13 CCV - Continuing calibration verification.

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- 3.14 CCVL – Low Level continuing calibration standard. Prepared at the same concentration as the Reporting limits for each analyte.
- 3.15 CCB - Continuing calibration blank.
- 3.16 LCS - Laboratory Control Sample -A quality control sample containing known concentration of analytes that is taken through the entire digestion and analysis procedure.
- 3.17 Calibration Blank - A blank solution containing 6% HNO₃ and 5% HCl for calibration.
- 3.18 Total Metals – The concentration determined on an unfiltered and acidified sample following vigorous digestion.
- 3.19 Dissolved or Soluble Metals – The concentration determined on a sample after passing through a 0.45um membrane filter, typically at the time of sample collection. Acidification and digestion are performed after filtration.
- 3.20 ELGA water – Reagent water that is deionized, filtered, and has a resistivity of 18 MΩcm⁻¹
- 3.21 LLQC- Lower limit of quantitation- Digested and analyzed to confirm the lowest quantitation limit
- 3.22 MB- Method Blank – A blank sample that is taken through each step of the analytical procedure, including the digestion procedure if it is used.

4.0 Interferences

There are four main types of interferences which affect ICP-OES: spectral, physical, chemical and memory interferences.

4.1 Spectral Interferences

These types of interferences are caused primarily from the overlap of elemental lines and background contributions. Interferences from spectral overlap are eliminated by the use of interfering element correction factors (IECs). Interferences caused by background contributions are eliminated by the use of background correction.

4.2 Physical Interferences

These types of interferences are caused by differences in the physical between the standards and the sample matrix. The major source of these interferences is a high

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dissolved solids concentration in a sample. Physical interferences are minimized by using an internal standard, diluting the samples and/or performing the method of standard addition.

Additionally, high salt concentrations can cause a buildup of salt at the tip of the nebulizer. This effect can be reduced by use of an argon saturator and/or a V-Groove nebulizer designed for high dissolved solid use.

4.3 Chemical Interferences

These are generally caused by molecular compound formation, ionization effects, and solvent evaporation effects. These effects can be minimized by careful selection of the operating conditions, by sample dilution, by buffering the sample, or by standard addition procedures. At TestAmerica Buffalo, an internal standard technique, which involves adding yttrium and indium that are both not found in the samples and verified to not cause an interelement spectral interference to the samples, standards, and blanks is used to minimize the ionization effects of the high level of easily ionized elements such as K and Na. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

4.4 Memory Interferences

Memory interferences (also referred to as carryover) result when analytes present in a sample contribute to the signals measured in one or more following samples. To minimize memory effects, appropriate rinse time must be allowed between all samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

- 4.5 The following tests may be performed to check for physical and chemical interferences. A serial dilution and a post-digestion spike is performed on a representative sample from each sample batch. The sample batch does not exceed twenty samples (MS/MSD count towards the twenty).

4.5.1 Serial Dilution (SD)

A serial dilution (1:5 dilution) is performed on a representative sample of each matrix of each sample group. If the analyte concentration is high enough, the serial dilution must agree within 10% of the original sample. If the serial dilution is outside the 10% limit, a chemical or physical interference effect is suspected.

4.5.2 Post-digestion Spike (PDS)

A post-digestion spike is performed on a representative sample within the sample group (client job) is spiked. Generally, the spike is performed on the same sample as the one on which the serial dilution is performed, unless there is limited volume.

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Spiking a sample consists of adding a specified amount of spike solutions to the unknown sample. Each spike solution contains various elements of interest.

4.5.3 The six spike solutions for Non-CLP samples are:

– **Spike 1 (Custom Inorganic Standard) Made by Ultra Scientific (ICUS-1370)**

This ULTRAgrade™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Antimony	40.0 µg/mL
Arsenic	40.0 µg/mL
Beryllium	40.0 µg/mL
Cadmium	40.0 µg/mL
Chromium	40.0 µg/mL
Cobalt	40.0 µg/mL
Copper	40.0 µg/mL
Lead	40.0 µg/mL
Manganese	40.0 µg/mL
Molybdenum	40.0 µg/mL
Nickel	40.0 µg/mL
Selenium	40.0 µg/mL
Thallium	40.0 µg/mL
Vanadium	40.0 µg/mL
Zinc	40.0 µg/mL
Titanium	40.0 µg/mL
Calcium	2000.0 µg/mL
Iron	2000.0 µg/mL
Magnesium	2000.0 µg/mL

Matrix: 5% HNO₃ in water. All weights are traceable to NIST traceable weight.

NOTE: These concentrations may vary slightly different between different lots. Exact concentrations may be found in the Certificates of Analysis and in TALS. This NOTE is also applicable to Spike 2, Spike 3, Spike 4, and Spike 5.

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- **Spike 2 (Custom Inorganic Standard) Made by Ultra Scientific (ICUS-3097)**
This ULTRAgrade™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Barium	40.0 µg/mL
Boron	40.0 µg/mL
Aluminum	2000 µg/mL
Potassium	2000 µg/mL
Sodium	2000 µg/mL
Lithium	40.0 µg/mL
Strontium	40.0 µg/mL

Matrix: 5% HNO₃ in water. All weights are traceable to NIST traceable weights.

- **Spike 3 (prepared by lab)**

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Silver	10 µg/mL

Matrix 2% HNO₃ in water. See 7.10 for preparation.

- **Spike 4 (prepared by lab)**

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Tin	40 µg/mL

Matrix: 5% HNO₃ in water. See 7.11 for preparation.

- **Spike 5 (prepared by lab)**

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Silicon	2000 µg/mL

Matrix: 5% HNO₃ in water. See 7.12 for preparation.

- **Spike 6 (prepared by lab)**

<u>ANALYTE</u>	<u>TRUE VALUE</u>
Sulfur	2000 µg/mL

Matrix: 5% HNO₃ in water. See 7.12 for preparation.

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Table 17.6 lists the final concentration of each element spiked.

To prepare a post-spike, add 0.05 mL of Spike 1, Spike 2, Spike 3, Spike 4, Spike 5 , and Spike 6 to 9.70 mL of sample. Mix thoroughly and analyze.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual (CW-E-M-001) and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care should be taken in the handling and disposing of all standards and samples. See section 14.0 for procedures on the disposal of standard and sample waste.

The matrix of all prepared standards and samples is 6% HNO₃, 5% HCl by volume. Preserved metals samples contain 1-2% HNO₃ and have a pH < 2. Gloves must be used when handling all standards and samples. Safety glasses and lab coats must be worn at all times within laboratory areas. Extra care should be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

The ICAP's plasma emits strong UV light and is harmful to vision. **AVOID LOOKING DIRECTLY AT THE PLASMA.**

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5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheets (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

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

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6.0 Equipment and Supplies

6.1 Instrumentation

Thermo Scientific ICAP 6500 ICP-OES Spectrometer, equipped with an ESI SC autosampler, computer, printer, and source of Argon gas. There are two ICAP Analyzers at TestAmerica Buffalo. They are designated as ICAP 1 and ICAP 2.

- o Spare parts for the ICAP:
 - nebulizers
 - torches
 - spray chambers
 - rotors
 - stators
 - fast switches
 - duo radial plasma view window
- o White/White or Gray/Gray pump tubing (drain)
- o Orange/Green pump tubing (IS)
- o Black/Black pump tubing (carrier)
- o Internal Standard tubing mixing kit (green-T)
- o Autosampler, Carrier, and Internal Standard probes
- o Sample loading coil

6.2 Supplies

- ✎ Volumetric flasks and graduated cylinders in various sizes from 50 mL to 2000 mL. These are used for standard preparation and sample dilution.
- ✎ Eppendorfs in various sizes. These are used for standard and sample preparation. The Eppendorfs and re-pipettors are verified using an analytical balance on a daily basis. They are calibrated on a quarterly basis along with the re-pipettors. An electronic spreadsheet contains the calibration results. At least one Eppendorf in each of the following ranges are used:
 - 10 µL → 100 µL
 - 50 µL → 200 µL
 - 50 µL → 250 µL
 - 100 µL → 1000 µL
 - 500 µL → 2500 µL
 - 2000 µL → 10000 µL

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- Disposable polypropylene pipette tips for the Eppendorfs in various sizes.
- Disposable 17x100 mm polypropylene culture tubes used for samples in the autosampler.
- 50 mL sample vials used for calibration and control standards in the autosampler.
- Repipettors and bottles for dispensing acids and blank for dilutions.
- Parafilm for covering some samples and standards when not in use.

7.0 Reagents and Standards

- 7.1 All standards and samples are prepared using 18 MΩcm⁻¹ ELGA water. The metals lab has an ELGA water system attached to a deionized water system. The ELGA water is monitored daily by the Wet Chemistry department and maintenance is performed as needed.
- 7.2 All standards are prepared with class A volumetric flasks, and calibrated Eppendorf pipettes.
- 7.3 All standards and samples are prepared with Trace Metals Grade Nitric and Hydrochloric Acids.
- 7.4 All the working standards and samples are prepared in the same matrix containing 6% HNO₃ and 5% HCl (by volume). All standards and samples are prepared such that the matrix is matched.
- 7.5 Standards are prepared as needed (about every 7-10 days for Calibration Standards).

Table 17.7 lists all purchased reagents and stock standards that are used. All purchased stock standards and solutions are certified by the manufacturer and the certificates kept on record. All stock and prepared solutions are logged into the LIMS.

The multi-element calibration standards and other solutions required (except for those used for quality control ICVs and CCVs) are prepared from stock solutions purchased from ULTRA SCIENTIFIC and INORGANIC VENTURES. The standard solutions used to prepare ICVs and CCVs purchased from HIGH PURITY or CPI. The use of two vendors of different lots ensures a second source verification of standards.

- 7.6 There are two types of solutions that are prepared from the purchased stock standards. They are prepared stock solutions and the working standards. Prepared stock solutions are used as intermediate standards for preparing the working standards. All prepared stock solutions and working standards are documented in the LIMS and are labeled with their name, preparation date, expiration date, and the initials of the analyst preparing the solution.

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They expire after six months or when the original starting stock standards expire, whichever is earlier.

The following information is recorded in the LIMS for each standard or solution:

- Name or concentration of the solution
- Preparation date
- Name or Initials of analyst preparing the solution
- The manufacturer of the starting stock solution
- The lot number of the starting stock solution
- The name or concentration of the starting stock solution
- The volume of the starting stock solution used
- The final volume of the solution being prepared
- The source acid or blank solution used

7.7 Blank solutions contain 6% HNO₃ and 5% HCl in ELGA water. The blank solution is used for the following:

- Calibration Blank
- ICB and CCBs
- Sample dilutions
- Preparation of matrix matched solutions and standards
- Instrument rinse and carrier

7.8 The Blank Solution is prepared by adding 1500 mL concentrated HNO₃ and 1250 mL concentrated HCl to a 25 liter plastic carboy half filled with ELGA water. Bring up to volume with ELGA water. This procedure may be scaled up or down. Use a graduated cylinder to add the acids. Be extremely careful when handling concentrated acids in these amounts (work in the fume hood wearing lab coat, gloves, and safety glasses).

7.9 The following spike solutions are prepared from ULTRA SCIENTIFIC single element stock standards: Spike 3, containing 10 µg/mL Ag; Spike 4, containing 40 µg/mL Sn; Spike 5, containing 2000 µg/mL Si, and Spike 6, containing 2000 µg/mL S .

7.10 Spike 3 (or Ag Spike), containing 10 µg/mL Ag, is prepared by adding 2.0 mL of 1,000 µg/mL Ag stock standard to a 200 mL volumetric flask half filled with 2% HNO₃ Blank Solution. Bring to the final volume with 2% HNO₃ Blank Solution. This spike is used for the post-spike.

7.11 Spike 4 (or Sn Spike), containing 40 µg/mL Sn, is prepared by adding 8.0 mL of 1,000 µg/mL Sn stock standard to a 200 mL volumetric flask filled with blank solution. Bring up the final volume with Blank solution. This spike is used for the post-spike.

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- 7.12 Spike 5 (or Si Spike), containing 2000 $\mu\text{g/mL}$ Sn, is prepared by adding 40.0 mL of 10,000 $\mu\text{g/mL}$ Si standard to a 200 mL volumetric flask filled with blank solution. Bring up the final volume with Blank solution. This spike is used for the post-spike.
- 7.13 Spike 6 (or S Spike), containing 2000 $\mu\text{g/mL}$ S, is prepared by adding 40.0 mL of 10,000 $\mu\text{g/mL}$ S standard to a 200 mL volumetric flask filled with blank solution. Bring up the final volume with Blank solution. This spike is used for the post-spike.
- 7.14 The following calibration standards and quality control standards are prepared in the laboratory from custom stock standards:
- IC2 (calibration standard)
 - IC3 (calibration standard)
 - IC4 (calibration standard)
 - ICSA (interference check standard A)
 - ICSAB (interference check standard AB)
 - CRI / ICVL / CCVL (low level verification standard)
 - IS (internal standard)
- 7.14.1 IC2 is prepared by adding 20 mL of both CRI stock Spikes and 0.04 mL of Sulfur spike to a 2000 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in IC2.
- 7.14.2 IC3 is prepared by adding 100 mL of IC4 (Section 7.14.3) to a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in IC3.
- 7.14.3 IC4 is prepared by adding 5.0 mL ICUS-3098; 5.0 mL TA-23, 5.0 mL TA-21, 0.5 mL each of 1000 $\mu\text{g/mL}$ Ag, Sn; and 2.5 mL 10,000 $\mu\text{g/mL}$ Si, S to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in IC4.
- 7.14.4 The ICSA is prepared by adding 25.0 mL of Al, Ca, and Mg (10,000 mg/mL Ultra Scientific stock solution) and 10.0 mL of Fe (10,000 mg/mL Ultra Scientific) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.9 for concentrations of elements in the ICSA.
- 7.14.5 The ICSAB is prepared by adding 50.0 mL of ICSAB stock solution (ICUS-3482), 0.1 mL 1,000 $\mu\text{g/mL}$ Ag stock standard, and .05 mL S 10,000 $\mu\text{g/mL}$ to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.9 for concentrations of elements in the ICSAB.

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- 7.14.6 The Low Level Verification standard (CRI/ICVL/CCVL) is prepared by adding 20 mL of CRI stock standard, CRI stock standard 2 and 0.04 mL Sulfur stock to a 2000 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.12 for concentrations of elements in the CRI/ICVL/CCVL.
- 7.14.7 Internal Standard: The IS is prepared by adding 0.25 mL of 10,000 µg/mL Y stock standard, and 2.5 mL of 10,000 µg/mL In stock standard to a 1000 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. The final concentration of the Internal Standard is 5.0 µg/mL Y and 25 µg/mL In.
- 7.15 The following quality control standards are prepared in the laboratory from HIGH PURITY stock standards:
- 7.15.1 The Continuing Calibration Verification (CCV) is prepared by adding 10.0 mL CAL STD #2-R Solution A; 10.0 mL CAL STD.#2-R Solution B; 1.0 mL each of 1000 µg/mL Ag, and 1.0 mL of 1000 µg/mL Sn, and 5.0 mL of 10,000 µg/mL S to a 2000 mL volumetric flask half filled with Blank Solution. Bring up to volume with Blank Solution. See Table 17.10 for concentrations of elements in the CCV.
- 7.15.2 The Initial Calibration Verification (ICV) is prepared using the same stock as the CCV. It is prepared by adding 75.0 mL of the CCV to a 100 mL volumetric flask and bringing it up to volume with Blank Solution. . See Table 17.10 for concentrations of elements in the ICV.

8.0 Sample Collection, Preservation, Shipment and Storage

- 8.1 The maximum holding time for metals samples is 180 days from sample collection. Aqueous samples are preserved with nitric acid to a pH<2. Soil samples do not require additional preservation, but are kept refrigerated.
- 8.2 Samples are prepared by a digestion procedure in the digestion lab. The digestates are brought to the instrumental lab by the digestion analyst. The digestates are stored on a shelf in the instrumental lab. When analysis on the digestates is complete, the digestates are placed in a main sample storage area. The main storage area is located in the garage near the digestion lab. The main storage area is used to store the original total samples, dissolved samples, digestates, and TCLP extracts. Digestates are kept for a minimum of 3 months before disposal. For CLP work the digestates are stored for 365 days after delivery of the data package. CLP samples must be refrigerated at 4 degrees C from the time of collection until digestion.

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8.3 **Controlled Access Storage:** CLP samples require controlled access storage with strict Chain-of-Custody procedures. Digestates for these samples are obtained from and returned to the cooler custodian. The custodian maintains both the original samples and the digestates in the locked controlled access storage cooler.

8.3.1 For CLP, the original samples are retained for 60 days following delivery of the final report package.

8.3.2 For CLP, digestates are retained for 365 days before disposal.

8.4 Most total and dissolved samples are preserved in the field at the time of sampling, or preserved by sample control when they are received. When sample preservation is required by the laboratory analyst (typically in cases where samples require laboratory filtration prior to preservation), a comment listing lot numbers of the preservation acid (and filter used if applicable) is attached to the affected samples. Samples preserved by the laboratory analyst are held for 24 hours (TestAmerica best practice) prior to preparation.

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	HDPE	50 mLs	HNO ₃ , pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 + 2°C	180 Days	N/A

¹Inclusive of digestion and analysis.

9.0 Quality Control

**Refer to the TestAmerica Corporate Quality Assurance Plan for general information and more specific detail. Often project-specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than TestAmerica's QAP or the following criteria. The following criteria are subsequently presented as the minimum criteria of those criteria deemed applicable in the absence of project-specific DQO's.*

Overview: This section provides the guidelines of the quality controls that are used to determine if data are acceptable or not. Depending on the clients' requests and each specific protocol, some QC samples may not be prepared and/or analyzed to each job. Any observed deviations must be documented for future references. If the analyst cannot make a decision about the acceptability of data, the supervisor must be consulted and the resolution must be documented. If data are unusable, the samples must be re-digested and/or re-analyzed

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depending on the situation. To insure quality data, all intermediate and working standards are prepared from high quality certified stock standards. All stock and prepared standards and solutions are logged into the LIMS to insure traceability. Stock solutions are purchased as often as necessary to insure a fresh source.

9.1 **Sample QC** - The following quality control samples are prepared as appropriate with each batch of digested samples:

9.1.1 Method Blank (MB) - For each digestion batch, one method blank is prepared for every 20 samples or fewer. Section 9.1.8 summarizes method blank compliance criteria.

9.1.2 Laboratory Control Sample (LCS) - For each digestion batch of aqueous matrix samples, a LCS prepared for every 20 or fewer samples. Refer to section 9.1.8 for compliance criteria. If the LCS for an element is outside of control limits for an element, then all the samples in the batch requiring that element must be re-digested. See Table 17.6 for the concentrations of each element.

9.1.3 Standard Reference Material (SRM) - For each digestion batch of soil (or other non-aqueous) matrix samples, an SRM is prepared for every 20 or fewer samples. The certified values for each element vary by manufacturer lot; can be found in the Certificate of Analysis and in the LIMS. The acceptance limits for recovery are provided by the manufacturer. If the SRM for an element is outside of control limits for an element, then all the samples in the batch requiring that element must be re-digested.

9.1.4 Matrix Duplicate (DU or MD) - For CLP methods (or per client request), one matrix duplicate is performed per digestion batch. See section 9.1.8 for duplicate compliance criteria. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or re-digest. Generally Matrix Duplicate analysis is performed only for CLP samples.

9.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – Note: The MS/MSD/MD count as samples when counting the number of samples for a batch. For MCAWW (200.7), one Matrix Spike is required for every 10 samples (10%). Every batch of 20 samples will have two Matrix Spikes and two Matrix Spike Duplicates. For SW846 methods, one Matrix Spike is required for every 20 samples (5%). Every batch of 20 samples will have one Matrix Spike and one Matrix Spike Duplicate. See Table 17.6 for the concentrations of the matrix spikes for each element. See section 9.1.8 for criteria for spike recovery and precision. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or re-digest. If the recovery for an element is outside the control limits, matrix effect is suspected for digestion and/or the determination. Generally MSD is performed for SW-846 and MCAWW protocols. For MCP/RCP, MS and MSD are redigested and

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reanalyzed if recoveries are less than 30%. Narrate with confirmation if recoveries are still less than 30%.

9.1.6 Post Digestion Spike (PDS) - A post digestion spike is performed based on client requirements. It is performed on the base sample that has the MS associated with it, and is used to verify matrix effect on element recovery in the MS/MSD. The post spike recovery must agree within the limits specified in section 9.1.8. If the post spike for an element is outside the control limits, the matrix effect is suspected in the analysis. For CLP, a post-digestion spike is analyzed if the pre-digestion spike recovery is outside control limits and the sample result does not exceed 4 times the spike added.

9.1.7 Serial Dilution (SD) - A serial dilution is performed on the base sample in the batch of 20 that has a matrix spike. The serial dilution is a 1:5 (one part of the sample to four parts of blank solution). The serial dilution should analyze within 10% of the undiluted sample (provided the level of analyte in the diluted sample is quantitative), or matrix effects are suspected in the analysis.

9.1.8 Sample QC frequency and control limits summary:

Sample QC	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Reporting Limit (SW846); < 2.2x MDL (MCAWW) < CRDL (CLP)
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	80-120% recovery (SW846 and CLP); 85-115% recovery (MCAWW)
Matrix Spike (MS) ² and Matrix Spike Duplicate (MSD) ²	1 in 20 or fewer (SW846); 1 in 10 or fewer (MCAWW)	75-125% recovery (SW846 and CLP) 70-130% recovery (MCAWW)
Matrix Spike Duplicate (MSD) ² Matrix Duplicate (MD) ²	1 in 20 or fewer samples	RPD < 20% (duplicates)
Std Reference. Material (SRM)	1 in 20 or fewer samples	Specified by manufacturer on a per lot basis
Serial Dilution (SD)	1 per digestion batch	+/- 10% of original result
Post Digestion Spike (PDS)	1 per digestion batch	80-120% recovery (SW846) 85-115% recovery (MCAWW)

¹ An LCS Duplicate (LCSD) or LCDSRM is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract (ex. MCP/RCP).

² The base sample for MS/MSD is arbitrarily selected, unless specifically requested by a client. A Matrix Duplicate may be used in place of a Matrix Spike Duplicate for some methods

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- 9.2 **Instrument QC** - The following instrument quality control samples are analyzed with each analytical run:
- 9.2.1 Initial Calibration Verification (ICV) - The ICV is prepared from separate source standards than the calibration standards. The ICV is analyzed following instrument calibration. If the ICV is outside the control limits for an element, then the instrument must be recalibrated or that element cannot be used from that analytical run. The measured values must be within $\pm 10\%$ of the true value for CLP and method 6010, or within $\pm 5\%$ of the true value for method 200.7. The RSD must be within 3%. See Table 17.10 for the true values of the ICV.
 - 9.2.2 Continuing Calibration Verification (CCV) - The ICV is prepared from separate source standards than the calibration standards. The CCV is analyzed at a frequency of every ten samples and at the end of each analytical run, and verifies the continued accuracy of the calibration. If the CCV is outside the control limits for an element, the 10 samples before and after that CCV should be reanalyzed for that element. Sample results may be accepted when the CCV indicates a high bias for an element, provided the sample result is a non-detect for that element. See Table 17.10 for the true values of the CCV.
 - 9.2.3 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) - The ICB is analyzed following the ICV. CCBs are analyzed following each CCV. Instrument blank analysis results should be less than the laboratory reporting limit (RL). If the CCB is outside of control limits for an element, the 10 samples before and after that CCB should be evaluated. Sample results may be accepted when they are non-detect for that element, or when the result is greater than 10x the high bias in the CCB. Otherwise, the samples should be reanalyzed for that element.
 - 9.2.4 ICSA – See section 9.2.8 for recovery criteria for the ICSA standard. If the ICSA is outside of control limits for an element, that element cannot be used from that analytical run. See Table 17.9 for the true values of the ICSA. (RCP guidelines require ICSA be run at the beginning and end of run with their samples)
 - 9.2.5 ICSAB - After analyzing the ICSA, analyze an ICSAB. See section 9.2.8 for recovery criteria. If the ICSAB is outside of control limits for an element, that element cannot be used from that analytical run. See Table 17.9 for the true values of the ICSAB. (RCP guidelines require ICSAB be run at the beginning and end of run with their samples)
 - 9.2.6 Low Level Verification (CRI/ICVL/CCVL) – Analyzed at the beginning of each analytical run following the ICV/ICB. Analysis frequency and criteria for this standard varies by method and by client requirements. If the CRI/ICVL/CCVL is outside of control limits for an element, that element cannot be used for any affected samples. See Table 17.12 for true values of the CRI/ICVL/CCVL.

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9.2.7 Internal Standard (IS) - The internal standard counts are monitored for each analysis. The internal standard counts must fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank. If the internal standard fails to fall between 50 and 150 percent of the initial blank the data from that particular sample may not be used from that analytical run. Recalibrate and reanalyze the sample. Dilute the sample if necessary.

9.2.8 Instrument QC frequency and control limits summary:

Instrument QC	Frequency	Control Limit
ICV	Start of each analytical run following a calibration	90-100% for SW846, CLP 95-105% for MCAWW
ICB	Start of each analytical run following the ICV	+/- RL
CRI / ICVL / CCVL	- Start of each analytical run for all methods; - End of each analytical run for CLP, 6010C, or by client QAPP; - Every 20 samples for CLP; - Recommended every 10 samples (with each CCV/CCB) for 6010C	50-150% recovery for 6010B, MCAWW 70-130% recovery for 6010C, CLP
ICSA	- Start of each analytical run for all methods; - End of each analytical run for CLP, or by client QAPP; - Every 20 samples for CLP	80-120% recovery for spiked analytes +/- 2x RL for non-spiked analytes
ICSAB	- Start of each analytical run for all methods	80-120% recovery
CCV	Every 10 samples, and at the end of each analytical run	90-110% recovery
CCB	Every 10 samples, and at the end of each analytical run	+/- RL

9.3 IEC, IDL, LDR, and LLQC

9.3.1 Interelement Correction Factors (IECs) are determined quarterly for each ICAP. The following solutions containing interfering elements are prepared from Ultra Scientific 10,000 µg/mL individual element stock standards (except 1000 µg/mL for Sn), and analyzed with corrections turned off. False positive or negative results for other elements indicate an IEC calculation is necessary. The solutions are prepared and analyzed at the established linear dynamic ranges.

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To calculate the IEC factor, divide the false result for an element to be corrected by the actual reading of the interfering element.

Example Calculation: The following results are obtained after analyzing a 1000 µg/mL Fe solution: Fe = 1028.0 µg/mL and Cd = 1.21 µg/mL

The IEC factor for Cadmium would be: $\frac{1.21}{1028.0} = 0.00118$

- 9.3.2 Instrument Detection Limits (IDLs) are determined quarterly for each ICAP as described in SOP BF-ME-018. The IDL is the minimum concentration of an analyte that can be measured with a high degree of confidence that the analyte concentration measured on a specific instrument is greater than random instrument noise. IDLs for the ICAPs are analyzed and calculated as described in SOP BF-ME-018.
- 9.3.3 Linear Ranges (LDR) are determined quarterly for each ICAP as described in SOP BF-ME-018. The linear range is the highest concentration of an analyte that an instrument can measure within ±5% of the known value.
- 9.3.4 Lower Limit of Quantitation (LLQC) are verified quarterly by digesting and analyzing the low-level calibration verification standard within +/-30% of the true values.
- 9.4 Contingencies for Handling Out-of-Control or Unacceptable Data
- 9.4.1 Data is to be evaluated in accordance with SOPs BF-GP-012 and BF-ME-013. When an out of control situation occurs, the analyst must use his/her best judgment and use any available resources to determine the corrective action to be taken. The analyst may need to seek immediate assistance from the supervisor, laboratory director, project manager, QA personnel or other experienced members of the staff if he/she is uncertain of the proper course of action. The test may need to be stopped until the problem is corrected since the problem may be instrumental and not chemical. Out of control data will never be released without the approval of the Supervisor, QA Manager, or Laboratory Director.
- 9.4.2 In the event acceptable data cannot be obtained, a Job Exception Form must be filed with the project manager and the client notified.
- 9.4.3 If the calibration or initial calibration checks fail for any analyte(s), (i.e., correlation coefficient is lower than 0.998; ICV, ICVL and/or ICB are out of control limits; ICSA or ICSAB are outside of control limits), the analytical run should be terminated, problems must be solved, the instrument recalibrated, and the restarted. Otherwise, the analytical run cannot be used for the out of control analyte(s).

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- 9.4.4 If a CCV, CCVL, and/or CCB are out of control limits for any analytes, affected analytes in the 10 samples before and after that CCV and/or CCB must be reanalyzed with the following exceptions:
- Results may be accepted when the CCV or CCB indicates a high bias, and the affected analytes are less than the reporting limit (<RL).
 - Results may be accepted when the CCB indicates a high bias, and the affected analytes are greater than 10x the CCB result.
- 9.4.5 If the LCS or SRM do not meet criteria for any analyte(s), the batch must be reanalyzed. If the reanalysis still does not meet criteria, that batch must be re-prepared and reanalyzed for the affected analyte(s).
- 9.4.6 If the Method Blank (MB) fails for an analyte, but samples do not contain that analyte higher than the reporting limit or samples contain that analyte higher than 10x the Method Blank result, the data is acceptable. Otherwise, the batch must be re-prepared and reanalyzed for that analyte.
- 9.4.7 If the RPD for the MSD (or MD) is out of control limits, the data should be reviewed to determine cause. If redigestion and reanalysis are still out of limits, the sample might be inhomogeneous and the data should be reported with qualification. Refer to table 17.15 for RPD criteria.
- 9.4.8 If Post Spike or Serial Dilution are outside of control limits, matrix effects in determination are suspected.
- 9.4.9 If the LCS, Post Spike and Serial Dilution are within QC limits, but the MS and/or MSD are out of control limits, matrix interference can be assumed and corrective action is not required.
- 9.4.10 For CLP and MCP/RCP, if the percent recovery of the CRI/ICVL falls outside the control limits of 70-130% (50-150% for Sb, Pb, TI), the CRI/ICVL must be re-analyzed for the outlying analytes. The same is true if the ICVL falls outside the control limits of 50-150% for 6010B and 200.7, and if the ICVL/CCVL falls outside the control limits of 70-130% for 6010C.
- 9.4.11 If the internal standard counts for any analysis fall outside of 50-150% of the internal standard counts in the Calibration Blank (ICIS), recalibrate and reanalyze the affected sample/samples.
- 9.4.12 When a value is more negative than the analytes's Reporting Limit (RL), it is the laboratory's procedure to dilute the sample until the value is less negative than the Reporting Limit (RL), or it is a positive value. The dilution is performed to

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demonstrate lessened affects of the interference present, enabling the analyst to view if the element of interest is present. The value from the dilution will be reported.

9.4.13 Dilutions are required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted to a level at or below the working range. See tables 17.15 and 17.16 for interfering elements for each ICAP.

10.0 Procedure

The matrix of all standards and samples for ICP are acidic. Nitrile gloves must be worn when handling all standards and samples. Safety glasses must be worn at all times in the laboratory. Extra care will be taken when dispensing concentrated acids and are to be dispensed only in a fume hood.

10.1 Sample Preparation

10.1.1 All samples are checked for the proper preservation at time of sample receipt in the sample receiving area. If the samples were not preserved, they are acidified and held for 24 hours. A sticker is affixed to the sample bottles. The pH is rechecked prior to digestion/analysis following the 24 hour waiting period.

10.1.2 Refer to the following SOPs for sample preparation details: BF-ME-002, BF-ME-003, BF-ME-005, BF-ME-007, and BF-ME-008.

10.2 Calibration

The ICAP is automatically calibrated at the beginning of each analytical run (at least daily). A calibration summary report is included with each analytical run report. A blank (IC1) and three levels of standards (IC2, IC3, IC4) are used to obtain a linear calibration plot for each element. The correlation for each element must be 0.998 or greater. If the correlation is less than 0.998 for a particular element, then the data for that element may not be used from that particular analytical run. See Table 17.8 for concentrations of elements in the calibration standards.

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10.3 Sample Analysis

10.3.1 The following is a daily checklist for the operation of the ICAP 6500 analyzer.

1. Empty the instrument and autosampler drain waste, if necessary.
2. Refill the autosampler rinse, if necessary.
3. Refill the internal standard and carrier solution, if necessary.
4. Inspect the pump tubing. Tubing replacement is recommended for approximately every 36 hours of instrument operation time.
5. Change or clean the torch, spray chamber, and sample nebulizer, as necessary. These are cleaned by sonicating them in 2% nitric acid.
6. Check the argon gas pressure, if necessary.
7. Ignite the plasma.
8. Prepare the standards and QC samples as needed (standards should be kept covered or re-poured daily) and place in the appropriate locations in the autosampler. See Section 7.0 for standards preparation.
9. Create an autosampler sequence, and assign it a run file name, and enter IDs for all samples to be analyzed.
10. Run an auto peak, if necessary.
11. Prepare all samples for analysis and place them into the autosampler.
12. Start the analysis.
13. Review raw data results (on screen) for instrument, sample, and QC failures, and to assess the need for any reanalysis.
14. When the analysis is complete, generate and merge PDF reports for the analytical run log, calibration report, and all raw data.
15. Export required sample result data as appropriate for import into the LIMS.
16. Turn off the plasma unless performing an additional analytical run.
17. Import data to the LIMS and perform validation of the data.
18. Empty the samples and standards into an appropriate AN waste receptacle.

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10.3.2 Analysis Sequence: The calibration standards are automatically analyzed at the beginning of each analytical run.

10.3.2.1 Each Non-CLP, 200.7, 6010C or 6010B analytical run is typed in the following format:

ICV
ICB
ICVL
ICSA
ICSAB
CCV
CCB
CCVL

9 samples

CCV
CCB
CCVL

9 samples

CCV
CCB
CCVL

9 samples

CCV
CCB
CCVL

9 samples

CCV
CCB
CCVL

9 samples

CCV
CCB
CCVL
ICSA
ICSAB
CCV
CCB
CCVL

Client specific (QAPPs)

—
—
—
—

Run a CCV and CCB after every 10 samples and at the end of the analytical run. Run the CCVL, ICSA, and ICSAB at the beginning of the analytical run, and also at the end of the analytical run if required by the client/QAPP.

NOTE: To be compliant with all protocols and clients' particular requests, extensive QC samples are routinely prepared and run. However, not all

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these QC samples are required for a particular protocol. For example, the ending CCVL, ICSA and ICSAB are not required by SW-864 (6010B) and 40 CFR protocol. Therefore, a particular run may not include ending CCVL, ICSA and ICSAB if that procedure only involves standard SW-864 and 40 CFR protocols. This note is also applicable to CLP procedure.

10.3.2.2 Each CLP analytical batch is typed in the following format:

ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB

10 samples

CCV
CCB

7 samples
CRI
ICSA
ICSAB

CCV
CCB

10 samples

CCV
CCB

7 samples
CRI
ICSA
ICAB

CCV
CCB

Run a CCV and CCB after every 10 samples, and at the end of the analytical run. Run a CRI, ICSA, and ICSAB every 17 samples, and at the end of the analytical run, directly followed by a CCV and CCB.

10.3.3 Typing an Analytical Run

Enter the analytical run into the autosampler table according to the following steps:

- Open Analyst window, choose method
- Click sequence tab at bottom of window
- Create a new autosampler table, replacing the "S_" in autosampler sequence name during table creation.

Example sequence name: I1031711A

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Key: I1 = ICAP 1 (I2 = ICAP 2)
03 = month
17 = day
11 = the last two digits of the year
A = run # (A, B, C, etc.)

- Enter solution IDs and sample IDs into table

10.3.4 Auto Peak

An auto peak is performed at least once per week, or as needed, following these steps:

- Place carrier probe into IC4 solution and allow to aspirate
- In top toolbar of Analyst window, click "Instrument" and choose "Perform Auto Peak"
- Click run.
- When auto-peak is complete, replace probe into carrier solution bottle.

10.3.5 Preparing Samples for the Autosampler

Using the autosampler table printout, set-up the samples in the autosampler. Use the disposable polypropylene culture tubes. Pour the samples into the culture tubes and place in the autosampler.

For 'Total Metals' and 'Dissolved Metals', the samples consist of the digestates received from the metals preparation department.

10.3.5.1 To prepare post spikes, add the following amounts of each spike solution to 9.7 mL of sample:

50 μ L - Spike 1 (Section 4.5.3)
50 μ L - Spike 2 (Section 4.5.3)
50 μ L - Spike 3 (Section 4.5.3)
50 μ L - Spike 4 (Section 4.5.3)
50 μ L - Spike 5 (Section 4.5.3)
50 μ L - Spike 6 (Section 4.5.3)

Mix each post spike thoroughly and place in autosampler.

10.3.5.2 To prepare the 1:5 serial dilution, add 2.0 mL of sample to 8.0 mL of calibration blank.

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10.3.6 Starting an Analysis

Once the autosampler table has been prepared, the samples, standard and quality control samples have been placed in the autosampler, and the auto peak has been performed, you are ready to begin the analysis. Use the following steps to begin the analysis.

- Minimize iTEVA software, and open ESI SC Autosampler software. This window must remain open throughout the duration of the run. Click "Initialize Autosampler" button.
- Maximize iTEVA software, and in Sequence tab of Analyst window, click the "Initialize" button that will establish communication between the autosampler and the instrument.
- Turn vacuum on to rinse. Send autosampler probe to rinse prior to calibration, and then send "home."
- To begin calibration, click yellow triangle "play" button in top toolbar of Sequence tab.
- After calibration and the subsequent quality control standards, CCVs and CCBs can be set up to run automatically after every 10 samples.
- View data during the run on the "Analysis" tab of the Analyst window.
- If desired, click the check box to automatically shut down the plasma after the run is completed. This is located in the autosampler setup window.

10.3.7 Printing Analysis Data

The ICAP instrument will print data during an analysis directly to a .pdf file, which needs to be merged after run has been completed. A run log is created by generating a sequence report in the Publisher window, and exporting the file to Excel. It is then initialed by the data reviewer, and scanned to a .pdf file. A calibration report is also generated in the Publisher window, and exported to a .pdf file. The three .pdf files are then merged into a complete raw data file.

10.4 Validation of the Data

When the analytical run is complete, the data must be checked for compliance with the method. Using Section 9.0 - Quality Control - check all the quality control samples (ICV, ICB, CCV's, CCB's, ICSA, ICSAB, IC Standards, CRI, CCVL, ICVL, and digested blank and LCS/LCSSRM) for compliance. If a quality control sample falls outside the required limits for an element, then that element must be rerun on another analytical run.

Also check the spikes and serial dilution for any matrix effects that might require a diluted sample run.

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11.0 Calculations / Data Reduction

Refer to sections 9.1.8 and 9.2.8 to determine if data are valid for each element. Any sample reading over the linear range must be diluted. Diluted samples must be run on required samples. Analyzing the sample and a series of spiked aliquots of the sample at different known concentrations performs an MSA.

- 11.1 The following calculations are illustrated:
 Relative Percent Difference (RPD) (See Section 11.1.1).
 Post spike calculation (See Section 11.1.2).
 Method of Standard Addition (MSA) calculation (See Section 11.1.3).

$$\% RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

- 11.1.1 The formula for calculating the relative percent difference is: Where,
 RPD = relative percent difference
 D₁ = first sample value
 D₂ = second sample value (replicate)

Sample calculation: A sample gave a reading of 2.51 µg/mL and the replicate reading was 2.39 µg/mL.

$$\% RPD = \frac{(2.51 - 2.39)}{(2.51 + 2.39) / 2} \times 100$$

$$RPD = 4.90\%$$

- 11.1.2 The formula for calculating the post spike recovery is:

$$\% Recovery = \frac{S_2 - S_1}{SA} \times 100$$

Where,
 S₂ = the post spiked sample reading
 S₁ = the sample reading
 SA = the spike added

Sample Calculation: A sample gave a reading of 0.250 µg/mL. The sample was post spiked with 2.000 µg/mL and gave a reading of 2.289 µg/mL.

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$$\% Recovery = \frac{2.289 - .250}{2.000} \times 100$$

$$\% Recovery = 102.0\%$$

11.1.3 The formula for calculating the simplest version of MSA (single-addition method) is:

$$C_x = \frac{S_A V_S C_S}{(S_B - S_A) V_x}$$

Where,

S_B = the concentration of the spiked sample

S_A = the concentration of the unspiked sample

V_S = volume of spike solution added.

C_S = concentration of spike solution

V_x = volume of sample before adding spike

C_x = the unknown sample concentration

Sample calculation: A sample gave a reading of 0.792 µg/mL. 50 µL of a 200 µg/mL spike solution was added to 10.0 mL of the sample. The spiked sample reading was 1.512 µg/mL.

$$C_x = \frac{(0.792)(0.05)(200.0)}{1.512 - 0.792(10.0)}$$

$$C_x = \frac{7.92}{7.20}$$

$$C_x = 1.10 \text{ ppm}$$

12.0 Method Performance

This SOP is applicable to digested sample matrices and soluble water samples.

12.1 Extensive quality control is used to insure compliance with method 6010B, 6010C, 200.7 and CLP protocol.

12.2 Thorough documentation is employed to insure traceability of reagents and standards.

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12.3 Approximate detection and reporting limits for ICAP 1 and ICAP 2 are found in Tables 17.2 and 17.3.

12.4 Samples that read above the instrument's linear range must be diluted.

12.5 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency. Refer to SOP BF-QA-001: Determination of Method Detection Limits.

12.6 Demonstration of Capabilities

Reference the corporate QA Manual. All employees analyzing the methods listed in the sop have documented Initial demonstration of capabilities, as well as demonstration of capabilities each year after. This documentation is forwarded to QA for approval and record keeping.

12.7 Training Requirements

The QA Manual or a Training SOP may be referenced for training requirements. If applicable, state required concentration of samples prepared for Precision and Accuracy study or alternate training procedure.

13.0 Pollution Control

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

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference AWM-HAZ.MG-01. The

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following waste streams are produced when this method is carried out.

- 14.1 All acidic waste consisting of sample and rinse solution: Dispose of as HNO₃ waste in an "AN" waste container.

15.0 References / Cross-References

- 15.1 Method 6010B Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010C Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update IV, Revision 3, February 2007.
- 15.2 ICAP 6500 Analyzer Operator's Manual.
- 15.3 ILM04.1, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.
- 15.4 ILM05.2, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.
- 15.5 ILM05.3, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.
- 15.6 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3.3, 40CFR Part 136, Appendix C, April 1991. (Approved for CWA compliance testing)
- 15.7 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Couple Plasma-Atomic Emission Spectrometry", Revision 4.4, US EPA / EMSL, May 1994. (Approved for SDWA compliance testing)

16.0 Method Modifications:

Item	Method xx	Modification
1	3005A	Adopted prep method for preparation of water samples for 6010B, 6010C and 200.7. See SOP BF-ME-002 for modifications.

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17.0 Attachments

- 17.1 Elements which are analyzed on ICP's
- 17.2 Approximate Water Detection Limits for the ICAP 6500 Analyzers
- 17.3 Approximate Soil Detection Limits for the ICAP 6500 Analyzers
- 17.4 Wavelengths and Background Points Used for Each Element on the ICAP 6500 Analyzer
- 17.5 Approximate Linear Dynamic Range of Each Element on the ICAP 6500 Analyzer
- 17.6 Concentration of each analyte for LCS, SRM, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike.
- 17.7 Reagents and Stock Solution which are purchased as Starting Materials for Preparation of Trace Standards
- 17.8 Concentration of Calibration Standards
- 17.9 Values for ICSA and ICSAB
- 17.10 Values for CCV and ICV
- 17.11 CLP Contract Required Detection Limits (CRDLs)
- 17.12 Concentration of Each Element in the CRI/CCVL/ICVL solution
- 17.13 Example of Batch Sheet for Metals
- 17.14 Certificates of Analysis for Custom Blend Standards
- 17.15 Interfering elements for ICAP 1
- 17.16 Interfering elements for ICAP 2

Revision History

- **Revision 8, Dated February 1, 2016**
 - Section 3.22 Added definition of MB
 - Section 4.5 Included MS/MSD count as samples
 - Section 7.14.1 Final volume was fixed to be 2000mL instead of 200mL
 - Section 7.14.4 Changed Fe amount from 25mL to 10mL
 - Section 7.14.5 Changed S amount from 0.01mL to 0.05mL
 - Section 7.14.6 Added CRI stock 2 and changed amount of CRI stocks from 5mL to 20mL and changed S amount from 0.01mL to 0.04mL and final volume from 500mL to 2000mL
 - Section 7.15.1 Changed final volume from 1000mL to 2000mL
 - Section 9.1.5 Added note about MS/MSD/MD count as samples in a batch
 - Section 9.4.10 Changed LLICV/LLCCV to ICVL/CCVL
 - Section 9.4.11 Changed internal standard to calculate from the ICB to the calibration blank (ICIS)
 - Section 10.3.2 Removed 200.7/6010B analytical sequence. No longer any difference from 6010C. Removed CRI.
 - Section 10.3.2.1 Changed from 10 to 9 samples between CCV,CCB,CCVL
 - Section 10.3.5.1 Changed amount of sample from 9.75mL to 9.5mL and added 50uL of Spike 6

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- Table 17.2 Removed 200.7 PQLs since 200.7 and 6010 PQLs are the same
- Table 17.3 Updated Soil PQLs for Li and Ag
- Table 17.5 Updated LDRs to current Jan 2016 LDRs
- Table 17.12 Updated CRI concentrations to match ICVL/CCVL
- Changed Laboratory Director, signature added
- **Revision 7, Dated Jan 13, 2015**
 - Section 1.3.2 Added Sulfur to table
 - Section 3.9 Changed LLICV to ICVL
 - Section 3.14 Changed LLCCV to CCVL
 - Section 4.5.3 Changed 5 spikes to 6 and added Sulfur spike info
 - Section 4.5.4 Removed CLP Spike info
 - Section 7.8 Changed HNO₃ and HCL volumes added
 - Section 7.10 Changed spike and volume used
 - Section 7.11 Changed spike and volume used
 - Section 7.12 Changed spike and volume used
 - Section 7.13 Added section for sulfur spike preparation
 - Section 7.14.1 Changed IC₂ preparation
 - Section 7.14.4 Changed ICSA preparation info
 - Section 7.14.5 Changed ICSAB Preparation info
 - Section 7.14.6 Changed CRI/CCVL preparation info
 - Section 7.15.1 Changed CCV preparation info
 - Section 9.4.4 Changed LLCCV to CCVL
 - Section 10.3.2.2 Changed LLCCV to CCVL
 - Table 17.2 Changed detection limits to include 6010 and 200.7 and add Sulfur
 - Table 17.4 Added Sulfur wavelength
 - Table 17.5 Added Sulfur LDR
 - Table 17.8 Changed concentration of IC₂ to match CCVL and add Sulfur
 - Table 17.9 Added Sulfur Concentration in the ICSAB
 - Table 17.10 Added Sulfur concentration
 - Table 17.12 Added concentration for both CRI and CCVL
- **Revision 6, Dated October 23, 2013**
 - Section 3.14 Removed BS
 - Section 3.15 Removed BLK
 - Section 3.17 Removed Method of Standard Additions
 - Section 3.21 Added LLQC and description
 - Section 4.5.1 Changed SRD to SD
 - Section 4.5.3 For Spike 5 changed see 7.11 for preparation to 7.12
 - Section 4.5.4 Renumbered CLP Spikes from 4.5.3

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- Section 6.1 Added spare parts for ICAP (stators, rotors, fast switches, duo radial plasma view window)
 - Section 7.5 Inserted Multi-element calibration standards are purchased from Ultra Scientific and Inorganic Ventures.
 - Section 7.13.3 Changed how IC4 is made. Removed ICUS 575 and added 5mL ICUS 3098, 5mL TA-23, 5 mL TA-21
 - Section 7.13.5 Changed how ICSAB is made. 50 mL ICUS 3482 and 0.1 mL Ag stock standard
 - Section 9.1.1 Remove BLK
 - Section 9.1.4 Remove DU
 - Section 9.1.6 Remove PS
 - Section 9.1.7 Remove SRD
 - Section 9.2.4 Changed MCP to RCP
 - Section 9.2.5 Changed MCP to RCP
 - Section 9.3.1 Remove table and inserted IECs are ran at the same concentration as the established linear ranges
 - Section 9.3.4 Added LLQC and how to prepare and analyze
 - Section 9.4.9 Removed BS
 - 10.35.1 Changed 9.80 mL to 9.75 mL
 - Table 17.7 Added Inorganic Ventures and removed ICUS 575 from Ultra Scientific
- **Revision 5, Dated September 27, 2012**
 - Section 9.1.8 Added MCP/RCP to footnotes as an example for LCSD
 - Sections 9.24 and 9.25, added MCP criteria for ICSA and ICSAB
 - Section 9.4.10 Added MCP/RCP to CRI criteria
 - Section 9.1.5 Added MCP/RCP criteria for MS/MSD; changed MS/MSD criteria for MCAWW
 -
 - **Revision 4, Dated February 2, 2012**
 - Section 1.3.2 and 17.4: Removed “background points” from text and table heading
 - Section 4.5.3: Changed four spike solutions to five spike solutions
 - Section 9.1.2: Changed BS to LCS
 - Section 9.1.4: Changed DUP to DU
 - Section 9.3.1: Added 100 µg/mL As to IEC table
 - Section 9.4.12: Deleted “The original negative value will be entered unless the dilution results in a detection above the reporting limit.” and added “The value from the dilution will be reported.”
 - Section 9.4.13: added section on diluting sample for high levels of interfering elements.

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- o 10.3.5.1: Added Spike 5
- o 10.3.7: Changed Excel to Open Office.org Calc
- o 17.15 and 17.16: Added Interfering element tables.
- **Revision 3, Dated June 03, 2011**
 - o Revised throughout for inclusion of additional analytes: Lithium (Li), Strontium (Sr), and Silicon (Si).
 - ICUS-574 (Spike 2) became ICUS-3097 (Li, Sr added)
 - ICUS-576 (Cal) became ICUS-3098 (Li, Sr added)
 - ICUS-1932 (CRI) became ICUS-3099 (Li, Sr, Si added)
 - ICUS-919 (ICSAB) became ICUS-3100 (Li, Sr, Si added).
 - All analyte information and concentration tables in sections 1 and 17 revised to include Li, Sr, and Si.
 - o Section 1.0: Reorganized and reworded Scope and Application Section
 - 1.3.2 Analyte table added, including lithium, strontium and silicon.
 - o Section 3.0: Removed section 3.21.
 - o Section 4.0: Removed reference to lithium nitrate buffer – no longer used
 - o Section 4.5.3: added Li & Sr to Spike 2; added Spike 5 (for Si); Post Spike sample volume changed from 9.8 mL to 9.75 mL.
 - o Section 5.0: Section reorganized and renumbered, removed section 5.3.
 - o Section 6.0: Section changed to pertain to new instrumentation.
 - o Section 7.0: Removed section 7.8.1 (redundant)
 - Removed section 7.12 – no longer use lithium nitrate
 - Removed 7.13.6 and 7.13.7 – no longer needed
 - o Section 7.9: Spike 5 added (for Si).
 - o Inserted Section 7.12 for Spike 5 preparation – subsequent section 7.x incremented accordingly.
 - o Section 8.0: Section reorganized and re-worded to reflect current practices.
 - Section 8.1 Soils are refrigerated.
 - Section 8.2 Digestates are kept for a minimum of 3 months
 - Section 8.3 Removed cool preservation from water section of chart.
 - o Section 9.0: Section reorganized and updated to reflect TestAmerica Method SOP Template.
 - Added requirements for 6010C.
 - o Section 9.2 correlation requirement changed from .995 to .998.
 - o Added lab procedure for diluting a negative value in section 9.4.12.

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- o Section 10.0: Updated to reflect new instrumentation and 6010C.
 - Deleted sections 10.2.2 through 10.2.6, and sections 10.3.1 through 10.3.7 – from old ICPs.
 - o Section 12.0: Replaced references to Trace #1 and Trace #2 with ICAP 1 and ICAP 2, included 6010C requirements.
 - o Section 15.0: Added reference to ICAP 6500 Analyzer Operator's Manual.
 - o Section 17.0: Removed 17.11 through 17.15 – old ICPs or redundant information.
 - o Section 17.14: example certificates of analysis updated for new ICUS standards.
 - o Throughout SOP: New LIMS Nomenclature
 - Cal1 became IC1
 - Cal2 became IC2
 - Cal3 became IC3
 - Cal4 became IC4
 - LCV became CRI
 - IFA became ICSA
 - IFB became ICSAB
 - BLK became MB
 - BS became LCS
 - SRM became LCSSRM
 - PS became PDS
 - SRD became SD
 - o Throughout SOP: changed procedures and nomenclature to pertain to new instrumentation.
 - ICAP 1 and ICAP 2 replaced Trace #1 and Trace #2
 - o Changed Quality Manager, signature changed.
-
- **Revision 2, Dated January 18, 2010**
 - o Removed 3.8 ICL-HCV-The highest calibration standard re-run directly after calibrating the instrument.
 - o Section 4.5.1 Removed "such that the analyte in the diluted sample is at least a factor of 10 above the IDL"
 - o Section 4.5.3 Added in the Note under the Table "Current concentrations may be found in the binder of the Certificates of Analysis and also in Element."
 - o Section 6.1 Changed STL: Buffalo to TestAmerica Buffalo
 - o Section 7.6 Removed standard logbook and added in Element
 - o Remove 8.3 "Soluble samples are stored in the main storage area with the digestates and the original total samples. All samples taken from the storage area must be logged out in the sample custody logbook that

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is kept in the digestion lab. Samples are logged back in when complete. The main storage area is kept locked when unattended. The key to the storage area can be obtained from the sample control personnel and returned to them when finished."

- o Remove 9.3 "ICL-The ICL is the highest calibration standard that is analyzed after the instrument is calibrated."
- o Section 9.4 Removed "It is analyzed after the ICL."
- o Section 9.10 Changed LFB to BS. Changed Laboratory Fortified Blank to Blank Spike.
- o Section 9.11 Changed LCS to SRM. Changed Laboratory Control Sample to Sample Reference Material.
- o Section 9.21.5 Changed LFB and LCS to BS and SRM.
- o Section 9.21.9 Changed LCS to BS.
- o Section 10.3.1 Changed STL Buffalo to TestAmerica Buffalo
- o Section 10.3.6.2 Changed "a piece of parafilm" with the lid.
- o Section 10.4.1 Removed HCV
- o Section 10.4.2 Removed HCV
- o Section 16.0 Added method modification
- o Removed Tables 17.19 and 17.20
- o Table 17.4 Added nm next to wavelength
- o Section 2.3 Removed AFCEE reference
- o Section 10.4.1 removed AFCEE references
- o Section 12.4 removed USACE reference
- o Section 7.6 changed 'standards logbook' to LIMS
- o Section 9.1 changed 'standards logbook' to LIMS
- o Section 10.3.6.5 rewords the procedure to clean the torch.
- o Section 17.7 removed ICUS-573 standard. No longer used

- **Revision 1, Dated July 07, 2009**

- o New LIMS nomenclature changes:
- o BLANK became CAL1
- o Std.1 became CAL2
- o Std.2 became CAL3
- o Std.3 became CAL4
- o Std.3 VER became HCV
- o CRI became LCV
- o ICSA became IFA
- o ICSAB became IFB
- o MBLK became BLK
- o LCS became SRM
- o LFB became BS
- o SD became MSD

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- o 10.3.6.12 Filling Argon Saturator: "Lower neb. pressure" deleted, replaced with "Turn plasma off." "Turn neb. pressure on" deleted, replaced with "Restart plasma."
 - o 10.4 Typing an Analytical Run: "SEQ-"added before all calibration and QC standards, names updated.
 - o 17.7 1,000 ug/ml Y replaced with 10,000 ug/ml Y under "From ULTRA SCIENTIFIC;" 10,000 ug/ml Y deleted from "From HIGH PURITY."
 - o 17.0 Example of a Data Review Summary Form for Metals replaced with an Example of a Bench Sheet for Metals.
 - o Metals Department manager change, signature.
- **Revision 0, Dated June 09, 2008**
 - o Integration for TestAmerica operations
 - o Quality Manager change, signature

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17.1 Elements Which are Analyzed on the ICAP 6500 Analyzer:

Aluminum	Al	Manganese	Mn
Arsenic	As	Molybdenum	Mo
Antimony	Sb	Nickel	Ni
Barium	Ba	Sodium	Na
Beryllium	Be	Potassium	K
Boron	B	Selenium	Se
Cadmium	Cd	Silicon	Si
Calcium	Ca	Silver	Ag
Chromium	Cr	Strontium	Sr
Cobalt	Co	Thallium	Tl
Copper	Cu	Tin	Sn
Iron	Fe	Titanium	Ti
Lead	Pb	Vanadium	V
Lithium	Li	Zinc	Zn
Magnesium	Mg	Sulfur	S

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17.2 Approximate Water Detection Limits for the ICAP 6500 Analyzers.

Element	Estimated IDL (mg/L)	Estimated MDL (mg/L)	Lab PQL (mg/L) 200.7/6010
Al	0.0563	0.060	0.2
Sb	0.0038	0.00679	0.02
As	0.0035	0.0055	0.015
Ba	0.00013	0.0005	0.002
Be	0.00027	0.0003	0.002
B	0.00114	0.004	0.02
Cd	0.00028	0.00033	0.002
Ca	0.0137	0.1	0.5
Cr	0.00062	0.00087	0.004
Co	0.00032	0.00063	0.004
Cu	0.00138	0.0015	0.01
Fe	0.0105	0.0193	0.05
Pb	0.0021	0.003	0.01
Li	0.0046	0.01	0.03
Mg	0.011	0.043	0.2
Mo	0.00045	0.00356	0.01
Ni	0.00091	0.00126	0.01
K	0.060	0.2	0.5
Se	0.0054	0.0087	0.025
Na	0.0920	0.324	1.0
Ag	0.00079	0.0017	0.006
Si	0.029	0.06	0.5
Sr	0.0003	0.001	0.005
Tl	0.0024	0.0102	0.02
V	0.0076	0.00108	0.005
Zn	0.00074	0.0017	0.01
Sn	0.00059	0.00505	0.01
Ti	0.00071	0.0011	0.005

Facility Distribution No. _____

Distributed To: _____

17.3 Approximate Soil Detection Limits for the ICAP 6500 Analyzers.

Element	Estimated IDL (mg/kg)	Estimated MDL (mg/kg)	Lab PQL (mg/kg)
Al	5.6	4.4	10.0
Sb	0.378	0.54	15.0
As	0.353	0.4	2.0
Ba	0.013	0.11	0.5
Be	0.027	0.028	0.2
B	0.114	0.19	2.0
Cd	0.028	0.03	0.2
Ca	1.366	3.3	50.0
Cr	0.062	0.2	0.5
Co	0.032	0.05	0.5
Cu	0.138	0.21	1.0
Fe	1.047	1.1	10.0
Pb	0.210	0.24	1.0
Li	0.460	1.0	30.0
Mg	1.11	.927	20.0
Mo	0.045	0.13	1.0
Ni	0.091	0.23	5.0
K	6.00	20.0	30.0
Se	.543	0.57	4.0
Na	9.20	13.0	140.0
Ag	0.079	0.2	0.5
Si	2.90	6.0	50.0
Sr	0.03	0.1	0.5
Tl	0.241	0.3	6.0
V	0.076	0.11	0.5
Zn	0.074	0.153	2.0
Sn	0.059	0.43	2.0

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Element	Estimated IDL (mg/kg)	Estimated MDL (mg/kg)	Lab PQL (mg/kg)
Ti	0.071	0.08	0.5
Mn	0.018	0.032	0.2

17.4 Wavelengths for Each Element on the ICAP 6500 Analyzer.

Element	Wavelength (nm)
Ag	328.068
Al	308.215
As	189.042
B	208.959
Ba	455.403
Be	313.042
Ca	317.933
Cd	228.802
Co	228.616
Cr	267.716
Cu	324.754
Fe	259.940 / 271.441
K	766.490
Li	670.784
Mg	279.079
Mn	257.610
Mo	202.030
Na	589.592 / 818.326
Ni	231.604
Pb	220.353
Sb	206.833
Se	196.090
Si	288.158
S	182.034
Sn	189.989
Sr	407.771

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Ti	334.904
Tl	190.856
V	292.402
Zn	206.200

17.5 Approximate Linear Dynamic Range of Each Element on the ICAP 6500 Analyzer.

Element	ICAP 1 (mg/L)	ICAP 2 (mg/L)
Al	1000	1000
Sb	50	50
As	15	15
Ba	10	10
Be	25	25
B	20	20
Cd	5	5
Ca	1500	1500
Cr	40	40
Co	20	20
Cu	25	25
Fe	800	800
Pb	120	120
Li	50	50
Mg	500	500
Mo	10	10
Ni	25	25
K	600	600
Se	60	60
Na	5000	5000
Ag	3	3
Si	100	100
Sr	10	10
Tl	40	40
V	50	50

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Element	ICAP 1 (mg/L)	ICAP 2 (mg/L)
Zn	20	20
Sn	4	4
Ti	50	50
Mn	125	125
S	50	50

17.6 Concentration of each analyte for LCS, SRM, Post-digestion Spike, matrix spike:

Element	LCS, Post-Digestion Spike and Non-CLP Matrix Spike (mg/L)		Soil Post-Digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Representative Soil LCS (mg/kg) changes per lot	
Al	10		200	16300	
Sb	0.20		40	117	
As	0.20		40	138	
Ba	0.20		40	269	
Be	0.20		40	157	
B	0.20		40	90	
Cd	0.20		40	71	
Ca	10		200	9660	
Cr	0.20		40	105	
Co	0.20		40	142	
Cu	0.20		40	110	
Fe	10		200	19100	
Pb	0.20		40	144	
Li	0.20		40	-	
Mg	10		200	4410	
Mo	0.20		40	90.4	
Ni	0.20		40	130	
K	10		200	5000	
Se	0.20		40	200	
Na	10.0		200	653	
Ag	0.20		40	45.1	
Si	10		200	-	

Facility Distribution No. _____

Distributed To: _____

Element	LCS, Post-Digestion Spike and Non-CLP Matrix Spike (mg/L)		Soil Post-Digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Representative Soil LCS (mg/kg) changes per lot	
Sr	0.20		40	246	
Tl	0.20		40	161	
V	0.20		40	67	
Zn	0.20		40	268	
Sn	0.20		40	160	
Ti	0.20		40	447	
Mn	0.20		40	539	

17.7 Reagents and Stock Solutions which are Purchased as Starting Materials for Preparation of Trace Standards.

From ULTRA SCIENTIFIC:

ICM-441

ICUS-3098

ICUS-3099

ICUS-3100

1,000 µg/mL Ag

10,000 µg/mL Al

10,000 µg/mL As

10,000 µg/mL B

10,000 µg/mL Ba

10,000 µg/mL Be

10,000 µg/mL Ca

10,000 µg/mL Cd

10,000 µg/mL Co

10,000 µg/mL Cr

10,000 µg/mL Cu

10,000 µg/mL Fe

10,000 µg/mL In *

10,000 µg/mL K

1,000 µg/mL Li

10,000 µg/mL Mg

10,000 µg/mL Mn

10,000 µg/mL Mo

10,000 µg/mL Ni

10,000 µg/mL Na

10,000 µg/mL Pb

10,000 µg/mL Sb

10,000 µg/mL Se

10,000 µg/mL Si

1,000 µg/mL Sn

10,000 µg/mL Sn

1,000 µg/mL Sr

10,000 µg/mL Ti

10,000 µg/mL Tl

10,000 µg/mL Y *

10,000 µg/mL Zn

Certificates of Analysis are attached for the custom blend standards listed as ICUS-(...) above.

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Distributed To: _____

From HIGH PURITY:

1,000 µg/mL Ag

1,000 µg/mL Sn

CAL STD #2-R Solution A

CAL STD #2-R Solution B

From CPI:

1,000 µg/mL V

From Inorganic Ventures

TA-23

TA-21

From JT-BAKER

Concentrated HCl (Trace Metals Grade)

Concentrated HNO₃ (Trace Metals Grade)

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Table 17.8 Concentrations of Calibration Standards: (in mg/L)

Element	IC2	IC3	IC4
Al	0.2	25.0	50.0
Sb	0.02	0.5	1.0
As	0.015	0.5	1.0
Ba	0.002	0.5	1.0
Be	0.002	0.5	1.0
Cd	0.002	0.5	1.0
Ca	0.5	25.0	50.0
Cr	0.004	0.5	1.0
Co	0.004	0.5	1.0
Cu	0.01	0.5	1.0
Fe	0.05	25.0	50.0
Li	0.03	0.5	1.0
Mg	0.2	25.0	50.0
Mn	0.003	0.5	1.0
Ni	0.01	0.5	1.0
Ag	0.006	0.5	1.0
Si	0.5	25.0	50.0
Sr	0.005	0.5	1.0
Tl	0.02	0.5	1.0
Zn	0.01	0.5	1.0
V	0.005	0.5	1.0
B	0.02	0.5	1.0
Mo	0.01	0.5	1.0
Ti	0.005	0.5	1.0
Sn	0.1	0.5	1.0
Se	0.025	0.5	1.0
Na	1.0	25.0	50.0
K	0.5	25.0	50.0
Pb	0.01	0.5	1.0
S	0.2	25.0	50.0

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Table 17.9 Concentrations for ICSA and ICSAB (in mg/L)

Element	ICSAB	ICSA
Al	500.0	500.0
Ca	500.0	500.0
Fe	100.0	200.0
Mg	500.0	500.0
Ag	0.2	-
As	0.1	-
Ba	0.5	-
Be	0.5	-
Cd	1.0	-
Co	0.5	-
Cr	0.5	-
Cu	0.5	-
Mn	0.5	-
Ni	1.0	-
Pb	0.05	-
Sb	0.6	-
Se	0.05	-
Tl	0.1	-
V	0.5	-
Zn	1	-
Li	0.5	-
Si	1.0	-
Sr	0.5	-
S	1.0	-

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Table 17.10 Concentrations for CCV and ICV (in mg/L):

Element	CCV	ICV
Al	25.0	18.75
Sb	0.5	0.375
As	0.5	0.375
Ba	0.5	0.375
Be	0.5	0.375
B	0.5	0.375
Cd	0.5	0.375
Ca	25.0	18.75
Cr	0.5	0.375
Co	0.5	0.375
Cu	0.5	0.375
Fe	25.0	18.75
Pb	0.5	0.375
Li	0.5	0.375
Mg	25.0	18.75
Mn	0.5	0.375
Mo	0.5	0.375
Ni	0.5	0.375
K	25.0	18.75
Se	0.5	0.375
Na	25.0	18.75
Ag	0.5	0.375
Si	25.0	50.0
Sr	0.5	0.375
Tl	0.5	0.375
V	0.5	0.375
Zn	0.5	0.375
Sn	0.5	0.375
Ti	0.5	0.375
Mn	0.5	0.375

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Element	CCV	ICV
S	25.0	18.75

Table 17.11 Contract Required Detection Limits (CRDL)

Analyte	CRDL (4.0) (ng/mL)	CRDL (5.0) (ng/mL)
Aluminum	200	200
Antimony	60	5
Arsenic	10	5
Barium	200	20
Beryllium	5	1
Cadmium	5	2
Calcium	5000	5000
Chromium	10	5
Cobalt	50	5
Copper	25	5
Iron	100	100
Lead	3	3
Magnesium	5000	5000
Manganese	15	10
Mercury	0.2	0.1
Nickel	40	20
Potassium	5000	5000
Selenium	5	5
Silver	10	5
Sodium	5000	5000
Thallium	10	5

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Vanadium	50	10
Zinc	20	10

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17.12 Element Concentrations in the CRI / ICVL/ CCVL Stock and Working Standards

Analyte	CRI	ICVL / CCVL
Aluminum	0.2	0.2
Antimony	0.02	0.02
Arsenic	0.015	0.015
Barium	0.002	0.002
Beryllium	0.002	0.002
Boron	0.02	0.02
Cadmium	0.002	0.002
Calcium	0.5	0.5
Chromium	0.004	0.004
Cobalt	0.004	0.004
Copper	0.01	0.01
Iron	0.05	0.05
Lead	0.01	0.01
Lithium	0.03	0.03
Magnesium	0.2	0.2
Manganese	0.003	0.003
Molybdenum	0.01	0.01
Nickel	0.01	0.01
Potassium	0.5	0.5
Selenium	0.025	0.025
Silver	0.006	0.006
Sodium	1.0	1.0
Silicon	0.5	0.5
Strontium	0.005	0.005
Thallium	0.02	0.02
Tin	0.01	0.01

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Titanium	0.005	0.005
Vanadium	0.005	0.005
Zinc	0.01	0.01
Sulfur	0.2	0.2

Facility Distribution No. _____

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17.13 Example of a Batch Sheet for Metals

Batch Number: 480-9051		Metals/Inorganics Analysis Sheet							Batch Open: 3/22/2011 9:50:00AM	
Method Code: 480-3005A_TOT-480		(To Accompany Samples to Instruments)							Batch End:	
Preparation, Total Metals										
	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments	Output Sample Lab ID
1	MB-480-9051/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		
2	LCS-480-9051/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		
3	480-2722-A-1 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		
4	480-2722-A-2 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		
5	480-2722-A-3 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		
6	480-2722-A-3-MS (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		
7	480-2722-A-3-MSD (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		
8	480-2743-G-1 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
9	480-2743-G-2 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
10	480-2743-G-4 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
11	480-2743-D-1 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
12	480-2743-D-2 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
13	480-2743-D-3 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		
14	480-2743-D-4 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		

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Distributed To: _____

Facility Distribution No. _____

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-9051

Analyst: Marzolf, Michelle

Batch Open: 3/22/2011 9:50:00AM

Method Code: 480-3005A_TOT-480

Batch End:

15	480-2743-D-5 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2	
16	480-2743-D-6 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2	
17	480-2743-D-7 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2	
18	480-2743-D-8 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2	
19	480-2746-E-1 (200.7)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2	
20	480-2724-O-1 (200.7)	2564	Water	50 mL	50 mL	3/23/11	10_Days - R	2	
21	480-2724-O-2 (200.7)	2564	Water	50 mL	50 mL	3/23/11	10_Days - R	2	
22	480-2326-A-11 (6010B)	N/A	Water	50 mL	50 mL	3/14/11	8_Days - R	1	
23	480-2326-A-12 (6010B)	N/A	Water	50 mL	50 mL	3/14/11	8_Days - R	1	

Printed : 3/22/2011

Page 2 of 7

TestAmerica Buffalo

Facility Distribution No. _____

Distributed To: _____

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-9051

Analyst: Marzolf, Michelle

Batch Open: 3/22/2011 9:50:00AM

Method Code: 480-3005A_TOT-480

Batch End:

	Input Sample Lab ID (Analytical Method)	(Sub-List)	Analytes	
1	MB 480-9051/1 N/A	N/A	N/A	
2	LCS 480-9051/2 N/A	N/A	N/A	
3	480-2722-A-1 (6010B)	(Local Method)	Pb	
4	480-2722-A-2 (6010B)	(Local Method)	Pb	
5	480-2722-A-3 (6010B)	(Local Method)	Pb	
6	480-2722-A-3 MS (6010B)	N/A	N/A	
7	480-2722-A-3 MSD (6010B)	N/A	N/A	
8	480-2749-G-1 (6010B)	(Local Method)	Ag, Al, As, Ba, Ca, Cd, Cr, Fe, K, Mg, Mn, Na, Pb, Se	
9	480-2749-G-2 (6010B)	(Local Method)	Ag, Al, As, Ba, Ca, Cd, Cr, Fe, K, Mg, Mn, Na, Pb, Se	
10	480-2749-G-4 (6010B)	(Local Method)	Ag, Al, As, Ba, Ca, Cd, Cr, Fe, K, Mg, Mn, Na, Pb, Se	
11	480-2743-D-1 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
12	480-2743-D-2 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
13	480-2743-D-3 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
14	480-2743-D-4 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
15	480-2743-D-5 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
16	480-2743-D-6 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	

3↑ CCV + CCB

Printed : 3/22/2011

Page 3 of 7

TestAmerica Buffalo

Distributed To: _____

Facility Distribution No. _____

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-9051

Analyst: Marzolf, Michelle

Batch Open: 3/22/2011 9:50:00AM

Method Code: 480-3005A_TOT-480

Batch End:

17	480-2743-D-7 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
18	480-2743-D-8 (6010B)	(TAL Metals ICP)	Ca, Fe, K, Mg, Mn, Na	
19	480-2746-E-1 (200.7)	(Local Method)	Cu	
20	480-2724-O-1 (200.7)	(Priority Pollutant Metals ICP)	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Sn, Ti, V, Zn	1:5 K, B, Na
21	480-2724-O-2 (200.7)	(Priority Pollutant Metals ICP)	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Sn, Ti, V, Zn	1:5 K+Na CCB T B B 10X
22	480-2326-A-11 (6010B)	(TAL Metals ICP)	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn	CCB T B
23	480-2326-A-12 (6010B)	(TAL Metals ICP)	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn	I

Analytes that are not being reported will be displayed in [...] brackets. Analytes that are not being reported but are on the spike list will be displayed in (...) parentheses.

I2032211B-4

I1032311A-2

MB B
LCS I

2724-1 1:5 B, K, Na

2724-2 1:5 K, Na

2326-11 B

2326-12 I

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17.14 Certificates of Analysis for Custom Blend Standards



Certificate of Analysis

RT00731
REC'D: 1/14/10
JAN

Inorganic Custom Standard

Catalog Number: ICUS-575

Lot Number: K00968

Job Number: J00010367

Lot Issue Date: 09/17/2009

Expiration Date: 10/31/2010

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value for each analyte is determined gravimetrically.

Analyte	True Value			Analytical Method
antimony	100.0	±	0.5 µg/mL	gravimetric
arsenic	100.0	±	0.5 µg/mL	gravimetric
beryllium	100.0	±	0.5 µg/mL	gravimetric
cadmium	100.0	±	0.5 µg/mL	gravimetric
chromium	100.0	±	0.5 µg/mL	gravimetric
cobalt	100.0	±	0.5 µg/mL	gravimetric
copper	100.0	±	0.5 µg/mL	gravimetric
lead	100.0	±	0.5 µg/mL	gravimetric
manganese	100.0	±	0.5 µg/mL	gravimetric
molybdenum	100.0	±	0.5 µg/mL	gravimetric
nickel	100.0	±	0.5 µg/mL	gravimetric
selenium	100.0	±	0.5 µg/mL	gravimetric
thallium	100.0	±	0.5 µg/mL	gravimetric
titanium	100.0	±	0.5 µg/mL	gravimetric
* vanadium	100.0	±	0.5 µg/mL	gravimetric
zinc	100.0	±	0.5 µg/mL	gravimetric
calcium	5009	±	25 µg/mL	gravimetric
iron	5007	±	25 µg/mL	gravimetric
magnesium	5002	±	25 µg/mL	gravimetric

Matrix: 5% nitric acid, trace hydrofluoric acid, and trace tartaric acid in low TOC water (< 50 ppb)

* light sensitive

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRA grade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001



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See Reverse For Additional Information

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Quality Assurance Manager

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Analytical Solutions

Certificate of Analysis

RT14762 R: 11/26/10 SMH
E: 9/30/12

CLP ICP Interference Check Standard #1

Catalog Number: ICM-441

Lot Number: J00734

Job Number: J00008573

Lot Issue Date: 08/05/2008

Expiration Date: 09/30/2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	True Value				Analytical Method	NIST SRM
aluminum	5009	±	25	µg/mL	ICP / ICP-MS	3101a
calcium	5005	±	25	µg/mL	ICP / ICP-MS	3109a
iron	2002	±	10	µg/mL	ICP / ICP-MS	3126a
magnesium	5002	±	25	µg/mL	ICP / ICP-MS	3131a

Matrix: 5% nitric acid in low TOC water (< 50 ppb)

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAGrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001

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AZI A
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Quality Assurance Manager

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Certificate of Analysis

Spike 2

Inorganic Custom Standard

Catalog Number: ICUS-3097

formally 574

Lot Number: M00419

Job Number: J00013015

Lot Issue Date: 04/21/2011

Expiration Date: 05/31/2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value at the 95% confidence level for each analyte is determined gravimetrically.

Analyte	True Value				Analytical Method
barium	40.00	±	0.20	µg/mL	gravimetric
boron	40.00	±	0.20	µg/mL	gravimetric
aluminum	2001	±	10	µg/mL	gravimetric
potassium	2001	±	10	µg/mL	gravimetric
sodium	2001	±	10	µg/mL	gravimetric
lithium	40.00	±	0.20	µg/mL	gravimetric
strontium	40.00	±	0.20	µg/mL	gravimetric

Matrix: 5% nitric acid in low TOC water (< 50 ppb)

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAGrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001



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Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-3098

formula 576

Lot Number: M00416

Job Number: J00013012

Lot Issue Date: 04/20/2011

Expiration Date: 05/31/2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value at the 95% confidence level for each analyte is determined gravimetrically.

Analyte	True Value				Analytical Method
barium	100.0	±	0.5	µg/mL	gravimetric
boron	100.0	±	0.5	µg/mL	gravimetric
aluminum	5005	±	25	µg/mL	gravimetric
potassium	5004	±	25	µg/mL	gravimetric
sodium	5002	±	25	µg/mL	gravimetric
* lithium	100.0	±	0.5	µg/mL	gravimetric
* strontium	100.0	±	0.5	µg/mL	gravimetric

Matrix: 5% nitric acid in low TOC water (< 50 ppb)

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAgrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.



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ISO 9001:2000
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Certificate of Analysis

TestAmerica Buffalo

SOP No. BF-ME-009, Rev.8

Effective Date: 02/01/2016

Inorganic Custom Standard

Catalog Number: ICUS-3099

Lot Number: M00385

Job Number: J00012948

Lot Issue Date: 04/14/2011

Expiration Date: 05/31/2012

Page No.: 63 of 69
080T

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value at the 95% confidence level for each analyte is determined gravimetrically.

Analyte	True Value	Analytical Method
aluminum	2.000 ± 0.010 µg/mL	gravimetric
antimony	0.2000 ± 0.0010 µg/mL	gravimetric
arsenic	0.1000 ± 0.0005 µg/mL	gravimetric
berium	0.0200 ± 0.0001 µg/mL	gravimetric
beryllium	0.0200 ± 0.0001 µg/mL	gravimetric
boron	0.2000 ± 0.0010 µg/mL	gravimetric
cadmium	0.0100 ± 0.00005 µg/mL	gravimetric
calcium	5.000 ± 0.025 µg/mL	gravimetric
chromium	0.0400 ± 0.0002 µg/mL	gravimetric
cobalt	0.0400 ± 0.0002 µg/mL	gravimetric
copper	0.1000 ± 0.0005 µg/mL	gravimetric
iron	0.5000 ± 0.0025 µg/mL	gravimetric
lead	0.0500 ± 0.00025 µg/mL	gravimetric
magnesium	2.000 ± 0.010 µg/mL	gravimetric
manganese	0.0300 ± 0.00015 µg/mL	gravimetric
molybdenum	0.1000 ± 0.0005 µg/mL	gravimetric
nickel	0.1000 ± 0.0005 µg/mL	gravimetric
potassium	5.000 ± 0.025 µg/mL	gravimetric
selenium	0.1500 ± 0.0008 µg/mL	gravimetric
* silver	0.0300 ± 0.00015 µg/mL	gravimetric
sodium	10.00 ± 0.05 µg/mL	gravimetric
thallium	0.2000 ± 0.0010 µg/mL	gravimetric
tin	0.1000 ± 0.0005 µg/mL	gravimetric
titanium	0.0500 ± 0.00025 µg/mL	gravimetric
* vanadium	0.0500 ± 0.00025 µg/mL	gravimetric
zinc	0.1000 ± 0.0005 µg/mL	gravimetric
*silicon	5.000 ± 0.025 µg/mL	gravimetric
*lithium	0.3000 ± 0.0015 µg/mL	gravimetric
*strontium	0.0500 ± 0.00025 µg/mL	gravimetric

Matrix: 5% nitric acid, trace tartaric acid in low TOC water (< 50 ppb)

* light sensitive

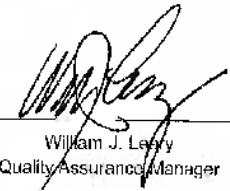
ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAgrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCCL Z-540-1 and ISO 9001.



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Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-3100

formerly 919

Lot Number: M00389

Job Number: J00012950

Lot Issue Date: 04/18/2011

Expiration Date: 05/31/2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value at the 95% confidence level for each analyte is determined gravimetrically.

Analyte	True Value			Analytical Method
* silver	2.000	±	0.010 mg/L	gravimetric
arsenic	1.000	±	0.005 mg/L	gravimetric
barium	5.000	±	0.025 mg/L	gravimetric
beryllium	5.000	±	0.025 mg/L	gravimetric
cadmium	10.00	±	0.05 mg/L	gravimetric
cobalt	5.000	±	0.025 mg/L	gravimetric
chromium	5.000	±	0.025 mg/L	gravimetric
copper	5.000	±	0.025 mg/L	gravimetric
manganese	5.000	±	0.025 mg/L	gravimetric
nickel	10.00	±	0.05 mg/L	gravimetric
lead	0.5000	±	0.0025 mg/L	gravimetric
antimony	5.000	±	0.025 mg/L	gravimetric
selenium	0.5000	±	0.0025 mg/L	gravimetric
thallium	1.000	±	0.005 mg/L	gravimetric
* vanadium	5.000	±	0.025 mg/L	gravimetric
zinc	10.00	±	0.05 mg/L	gravimetric
aluminum	5005	±	25 mg/L	gravimetric
calcium	5005	±	25 mg/L	gravimetric
iron	1001	±	5 mg/L	gravimetric
magnesium	5002	±	25 mg/L	gravimetric
* silicon	10.00	±	0.05 mg/L	gravimetric
* lithium	5.000	±	0.025 mg/L	gravimetric
* strontium	5.000	±	0.025 mg/L	gravimetric

Matrix: 5% nitric acid, trace hydrofluoric acid, trace tartaric acid in low TOC water (< 50 ppb)

* light sensitive

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAgrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001



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Ⓢ PECO 10/15/10 RT12812



Certificate of Analysis

SM-606-044 (CAL STD #2RR)

Solution A

Lot # 1027723

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
High Purity Metals, Salts, and Oxides	99.98+ %	HNO ₃ , 5%	µg/mL = 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: OCT 06 2011
MSDS ATTACHED

Facility Distribution No. _____

Distributed To: _____

P220 10/15/10

RT22813



Certificate of Analysis

SM-606-044 (CAL STD #2RR)

Solution B

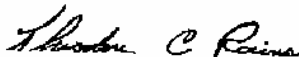
Lot # 1027724

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
High Purity Metals, Salts or Oxides	99.961 %	INO ₃ , 5% + Tr HF	100 µg/mL ± 0.5% Antimony Molybdenum Titanium

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.



Theodore C. Rains, Ph.D.
President

Exp Date: OCT 06 2011
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Certificate of Analysis

RT10384

REC'D: 8/24/10
AW

Inorganic Custom Standard

Catalog Number: ICUS-1370

Lot Number: L00948

Job Number: J00011904

Lot Issue Date: 08/17/2010

Expiration Date: 09/30/2011

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The certified value and uncertainty value for each analyte is determined gravimetrically.

Analyte	True Value			Analytical Method
antimony	40.00	±	0.20 µg/mL	gravimetric
arsenic	40.00	±	0.20 µg/mL	gravimetric
beryllium	40.00	±	0.20 µg/mL	gravimetric
cadmium	40.00	±	0.20 µg/mL	gravimetric
chromium	40.00	±	0.20 µg/mL	gravimetric
cobalt	40.00	±	0.20 µg/mL	gravimetric
copper	40.00	±	0.20 µg/mL	gravimetric
lead	40.00	±	0.20 µg/mL	gravimetric
manganese	40.00	±	0.20 µg/mL	gravimetric
molybdenum	40.00	±	0.20 µg/mL	gravimetric
nickel	40.00	±	0.20 µg/mL	gravimetric
selenium	40.00	±	0.20 µg/mL	gravimetric
thallium	40.00	±	0.20 µg/mL	gravimetric
* vanadium	40.00	±	0.20 µg/mL	gravimetric
zinc	40.00	±	0.20 µg/mL	gravimetric
titanium	40.00	±	0.20 µg/mL	gravimetric
calcium	2000	±	10 µg/mL	gravimetric
iron	2000	±	10 µg/mL	gravimetric
magnesium	2000	±	10 µg/mL	gravimetric

Matrix: 5% nitric acid, trace tartaric acid in low TOC water (< 50 ppb)

* light sensitive

ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles

in the manufacturing of ULTRAggrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCCL Z-540-1 and ISO 9001



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Quality Assurance Manager

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17.15: Interfering elements on ICAP 1

Interfering Analyte:	Interfered Analyte:	Interfering Analyte:	Interfered Analyte:
Al	Pb Se	Si	B Ba Cd, Co, Pb,
As	Cd	Ti	Be Co Pb Si Sn, Ti, V
Co	Ni Pb Ti	V	Al, Cd, Ti, Be
Cr	As Sb V Zn	Mo	B Co Pb
Fe	Cd Cr, Pb, V		
Mn	V		

Facility Distribution No. _____

Distributed To: _____

17.16: Interfering elements on ICAP 2

Interfering Analyte:	Interfered Analyte:	Interfering Analyte:	Interfered Analyte:
Al	Pb, Se	Si	B
As	Cd		Ba
Co	Cd		Cd
	Tl		Co
Cr	As		Pb
	Sb	Ti	Ag
	Ti		Be
	V		Co
	Zn		Cu
Fe	Ag		Pb
	Cr		Sn
	Ni		Tl
	Pb		V
	Sb	Ti	Ni
Mn	Tl	V	Al
	V		Ag
Mo	As		Be
	B		Cd
	Co		Cu
	Pb		Tl
	Sb		

Facility Distribution No. _____

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**Title: Mercury Preparation and Analysis
[Methods 245.1, 7470A, 7471A, 7471B]**

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Approvals (Signature/Date):



Todd Brandt
Department Manager

10/2/15

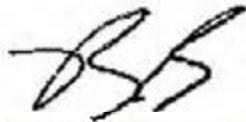
Date



Jennifer Pierce
Operations Manager

10/2/15

Date



Brad Prinzi
Quality Assurance Manager

10/2/15

Date



Kene Kasperek
Laboratory Director

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1.0 Scope and Application

- 1.1** This method is used for the determination of Mercury in aqueous and solid environmental samples. This procedure is used to analyze organic and inorganic mercury in drinking water, surface water, waste, and saline waters, both domestic and industrial wastes.
- 1.2** This method is based upon SW-846, 3rd edition methods 7470A, 7471A, 7471B and also conforms to the EPA Environmental Methods Management Council's "Guidelines and Format for Methods to Be Proposed at 40 CFR, part 136" (Method 245.1).
- 1.3** This method is for the determination of Hg by cold-vapor atomic absorption (CVAA) in the range of 0.2 µg/L to 10.0 µg/L. The range may be extended to higher levels by selection of a smaller sample size or by dilution of existing samples.
- 1.4** This method is used only by analysts experienced in the use of the chemical principles outlined in this SOP and who are trained thoroughly in the sample handling and instrumental techniques described in this method.
- 1.5** This method is "performance based." The laboratory is permitted to modify the method to overcome interferences or lower the cost of measurements provided all performance criteria are met.
- 1.6** On occasion, clients may request modifications to this SOP. These modifications are addressed following the procedures outlined in the lab Quality Assurance Manual (QAM).

1.7 Analytes, Matrix(s), and Reporting Limits

- 1.7.1** Total, Total Recoverable, and Dissolved (Soluble) Mercury.
- 1.7.2** This SOP is used for the preparation and analysis of groundwater, surface water, drinking water, TCLPs, leachates, filtered collection wastes, sand, rock, concrete, soil, sediment, and sludge samples.
- 1.7.3** Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error, client requirements, values specified by the EPA methods or other project and client requirements. Wherever possible, reporting is limited to values approximately 3–5 times the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. The reporting limit for mercury analysis in an aqueous matrix is 0.2 µg/L while the reporting limit for solid samples is typically 0.025 µg/g.

2.0 Summary of Method

- 2.1** Samples are digested first by oxidation of Hg in the samples to the Hg^{2+} oxidation state under strongly acidic and oxidizing conditions and near boiling temperatures. Potassium permanganate (KMnO_4) and potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$) (aqueous samples only) are added to aid in the oxidation of organic mercury compounds and to eliminate possible interference from sulfides and organic materials. Potassium permanganate is later reduced with hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) prior to the digestate being analyzed.
- 2.2** Analysis by cold-vapor atomic absorption is based on the absorption of radiation at the 253.7-nm wavelength by Hg vapor. The Hg^{2+} in a digested sample is reduced to the elemental state and aerated from solution in-line. The Hg vapor passes through an optical cell positioned in the light path of an atomic absorption spectrometer. Hg concentration is determined as a function of the measured absorption.
- 2.3** Quality is assured through the analysis of preparation blanks, laboratory control samples, matrix spikes, duplicates, and reference standards (solids).

3.0 Definitions

- 3.1 Total Mercury:** All oxidizable mercury forms and species found in an unfiltered aqueous or solid sample matrix. This includes, but is not limited to, Hg (0), Hg (I), Hg (II), strongly organo-complexed Hg (II) compounds, adsorbed particulate Hg, and several tested covalently bound organo-mercury compounds.
- 3.2 Dissolved (Soluble) Mercury:** All oxidizable mercury forms and species found in the filtrate of an aqueous solution that has been filtered through a 0.45 micron filter and then acidified to a $\text{pH} < 2$.
- 3.3** Any other definitions contained within this document are standard definitions as defined by the TestAmerica Buffalo Laboratory Quality Manual.

4.0 Interferences

4.1 Contamination

- 4.1.1 Contamination Control:** Any object or substance that contacts the sample should be mercury free and free from any material that may interfere with the analysis of mercury. Although contamination control is essential, personal health and safety remain the highest priority. Section 5 of this SOP gives suggestions and requirements for personal safety.
- 4.1.2 Avoiding Contamination:** The best way to control contamination is to completely avoid exposure of the sample to contamination in the first place. Avoiding exposure means performing operations in an area known to be free of any traces of mercury. Two of the most important factors in avoiding and/or reducing sample contamination are (1) an awareness of potential sources of contamination and (2) strict attention to the work being done
- 4.1.3 Minimize Exposure:** The apparatus and/or glassware that will come into contact with the samples, blanks, or standard solutions are to be opened or exposed only in a clean area of the lab. When any relevant materials, glassware or instruments are not being used, cover with a plastic liner or remove from the area of analysis to avoid accidental exposure.
- 4.1.4 Clean Work Surfaces:** Before a given batch of samples is processed, the analyst makes certain that all work surfaces in the hood, the bench and other areas are clean, thereby minimizing potential for contamination from previous batches.
- 4.1.5 Wear Gloves:** Sampling personnel wear clean, non-talc gloves during all operations involving handling of any instrument, glassware, samples or blanks. Only clean gloves may touch the instruments. If another object or substance is touched, the gloves must be changed before resuming work on the instrument. If it is suspected that gloves have become contaminated, work must be halted, the contaminated gloves removed, and a new pair put on. It is a good practice to change gloves between working on different sample matrices.
- 4.1.6 Use Mercury-Free Materials:** All materials used for the preparation and analysis of mercury at ambient water quality criteria levels must be non-metallic, free of material that may contain metals, or both. Mercury thermometers are not to be used within the mercury preparation or analysis areas.
- 4.1.7 Containers:** Each new container type is tested before use, because Mercury vapors can diffuse in or out of certain types of materials, resulting in results that are biased high or low.
- 4.1.8 Contamination from Reagents:** Contamination can be introduced into samples from the method reagents used during preparation and analysis. Reagents are monitored using method blanks included in each batch. When a reagent is suspected to be impure, it will be analyzed. If the blank is lower than MDL, that reagent can be used.

4.1.9 Contamination from Carryover: Contamination may occur when a sample containing a low concentration of mercury is analyzed immediately after a sample containing a high concentration of mercury. When an unusually concentrated sample (approximately 100+ ppb) is encountered, the cleaning (rinse) time is extended before proceeding with the next sample. To avoid this, samples that are known, or at least suspected of having the lowest mercury content should be analyzed first. As a guideline, samples with results *less than* 10x the RL which immediately follow a sample with a result greater than the LDR, should be reanalyzed to check for carryover.

4.1.10 Contamination from Samples (cross-contamination): Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates and other undiluted samples containing concentrations of mercury greater than 100 ppb are processed and analyzed. Samples known or suspected to contain Hg concentrations greater than 100 ppb should be diluted prior to bringing them into the laboratory whenever possible, or if prior dilution is not possible, the digestate should be diluted prior to analysis. Such samples should be handled with care to avoid contamination of other samples. Change gloves after handling samples known to contain high levels of mercury.

4.2 Chemical Interference

4.2.1 Any material which can absorb radiation at the 253.7-nm wavelength has the potential to cause a positive interference. Materials that inhibit the reduction of Hg^{2+} to Hg^0 , or which inhibit the aeration of Hg^0 into the vapor phase have the potential to cause a negative interference. The sample digestion procedure is designed to eliminate common interferences of these types.

4.2.2 The most common interferences come from brine samples and samples containing high levels of sulfides. Use of additional potassium permanganate can remove most of these interferences, however, very high levels can lead to low mercury recoveries. Other interferences include chlorides and iodides (halides), gold, or copper (reported at levels >10 ppm). High levels of organic solvents, such as acetone, hexane, alcohols, and glycols can also interfere.

4.3 Physical Interference

4.3.1 Physical interference can result from a damaged or dirty optical cell (including cracks, smudges, or condensed water vapor), and air bubbles trapped in samples or introduced in-line due to leaks in tubing or junctions.

4.3.2 Inconsistent levels of water vapor within the optical cell can result in instrument drift. Water vapor is regulated through use of a dehydrator; however excessive variations in atmospheric conditions surrounding the dehydrator can result in varying performance. Temperature changes greater than 3-5 °C can also result in instrument drift. For best performance, the sample delivery and detection system should be kept in as stable an operating environment as possible.

5.0 Safety

- 5.1** Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), and in this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this SOP to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2** The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined; however, each compound is treated as a potential health hazard. Exposure to these compounds is reduced to the lowest possible level.
- 5.3** The laboratory is responsible for maintaining a current awareness file of OSHA regulations for the safe handling of the chemicals specified in this procedure. OSHA rules require that a reference file of Safety Data Sheets (SDS) is made available to all personnel involved in these analyses. All SDS may be viewed on the Test America intranet.

5.4 Specific Safety Concerns or Requirements

- 5.4.1** This SOP uses several highly concentrated mineral acids, as well as strong oxidizers. Analysts must be familiar with proper response procedures for large and small spills, and for physical contact (see reference to CW-E-M-001 in sect 5.1). An acid spill kit is to be stored in a readily accessible location within the laboratory.
- 5.4.2** All digestion of acidified samples is to be conducted inside of a fume hood. The fume hood is periodically monitored to ensure its proper functioning and airflow requirements. This is especially important during soil digestion in which potassium permanganate can react with hydrochloric acid to produce chlorine gas.
- 5.4.3** Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH may react violently when acids are added. Use extra care and add acids slowly to leachates, colored samples, samples containing bubbles or foam, samples with swollen containers, or sample with strong odors.
- 5.4.4** Chronic mercury exposure may cause kidney damage, muscle tremors, spasms, personality changes, depression, irritability and nervousness. Organo-mercurials may cause permanent brain damage. Because of the toxicological and physical properties of Hg, only trained personnel familiar with handling mercury standards should handle standards.
- 5.4.5** As recommended, the laboratory purchases a dilute standard of Hg so that its use won't compromise the health and safety of the analyst. When samples known or suspected of containing high concentrations of mercury are handled, all operations are performed in a controlled area of the laboratory, preferably in a fume hood with adequate airflow and ventilation.

- 5.4.6** Mercury containing exhaust vapors leaving the instrument are passed through a column of activated carbon, filter trap containing gold or sulfur, or other suitable filter or trap in order to sequester mercury vapors away from the analyst.
- 5.4.7** While this procedure does call for the trace analysis of mercury at extremely small levels, it is still possible to be exposed to toxic levels of mercury during normal laboratory conditions. Mercury is at it most toxic when it is allowed to enter the bloodstream, therefore, any analyst who has an open wound or other such injury should take special care in avoiding mercury exposure.
- 5.4.8** The laboratory contains a mercury spill kit in case of serious mercury exposure. The kit is located in an area familiar to all that work in the laboratory, in the cabinet under the sink. Personnel can use mild soap with plenty of scrubbing in order to decontaminate skin. In the case of open wounds, professional help is to be sought immediately. All glassware, tools and surfaces are cleaned with sulfur powder in order to reduce any mercury present to non-volatile mercury sulfide. Washing the surface with reagent water will complete the cleansing process.

5.5 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (100 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (for Hg compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3- TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Potassium Permanganate	Oxidizer	5 Mg/M3 Ceiling (for Mn compounds)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 All equipment and supplies will be free of trace mercury, or at least at a level below the MDL of the method. All reusable equipment is cleaned according to the SOP BF-GP-003, Attachment 3, "Cleaning Procedure for Metals Glassware".

6.2 **Supplies and Equipment for Sample Preparation**

6.2.1 50 mL graduated Digestion Tubes and Caps, with (at a minimum) marks at 30 mL and 50 mL. If cup volumes are not certified by the manufacturer, the 30 mL and 50 mL marks must be verified on a per lot basis. Verify by filling 5 individual tubes with reagent water to the appropriate (30 mL and/or 50 mL) mark by eye level and weighed. The average weight of water in the five cups must agree to within 1%. The results are logged into a spreadsheet and stored on the network drive.

6.2.2 *Environmental Express* Hot Blocks capable of maintaining a temperature of 95°C (+/- 3°C). Hot Block temperature is verified daily prior to digestion and after digestion is complete. Temperatures are recorded in log book and batch.

6.2.3 Digestion tube racks to store and remove the samples from the hot blocks.

6.2.4 Bottle Repipettors for dispensing acids and reagents to samples.

6.2.5 Nalgene brand Wash Bottles for dispensing reagent water.

- 6.2.6 Thermometer covering a range of 0-150 °C. Thermometers are calibrated against a NIST certified thermometer in accordance with SOP BF-GP-020.
- 6.2.7 Analytical Balance accurate to ± 0.1 mg (currently a Denver P-214). Calibration to be verified daily. Balances are serviced yearly. (See SOP BF-GP-002)
- 6.2.8 Weigh boats and spatulas for soil samples and reagent preparation.
- 6.2.9 50 mL, 100 mL, and 2000 mL volumetric flasks (Class A) for preparation of reagents and standards.
- 6.2.10 *Eppendorf* Pipettes; varying volumes, preferably one for each volume to be dispensed. Minimally, pipettes in the following ranges are needed: 0.05-0.2 mL, 0.1-1.0 mL, 0.5-2.5 mL, and 2.0-10.0 mL. Pipettes are verified daily and calibrated quarterly in accordance with SOP BF-GP-001.
- 6.2.11 Time device for monitoring digestion step times.

6.3 Supplies and Instrumentation for Sample Analysis

- 6.3.1 15 mL test tubes for analyzing samples.
- 6.3.2 Mercury Adsorbent filter for the instrument exhaust line: currently used -- a mercury adsorbing activated carbon filter from *Perkin-Elmer*.
- 6.3.3 Pump tubing for delivery of samples and reagents to the instrument:
 - 6.3.3.1 Yellow-Blue (0.51 mm) tubing: for SnCl_2 introduction.
 - 6.3.3.2 Orange-Yellow (1.52 mm) tubing: for Sample introduction
 - 6.3.3.3 Green-Green (1.88 mm) tubing: for waste drain
- 6.3.4 Cold-Vapor Atomic Absorption (CVAA) instrument capable of detecting in the range of 0.2 $\mu\text{g/L}$ to 10.0 $\mu\text{g/L}$. Currently: (1) Leeman Labs PS200 II Automated Mercury Analyzer, and (1) Leeman Labs Hydra AA Automated Mercury Analyzer. Both instruments are operated using WinHg software version 1.1.
- 6.3.5 Various consumable and replacement instrument parts available and purchased from the instrument manufacturer.

7.0 Reagents and Standards

7.1 Reagents:

- 7.1.1 Laboratory Reagent Water: ($\text{DI H}_2\text{O}$); Deionized water from a purified source. Water will be monitored for Hg, especially after ion exchange beds are changed.
- 7.1.2 Silicon (IV) Oxide: (SiO_2); Used as a blank soil matrix substitute. High purity grade (typically 99.995% for metals).

7.2 Stock Acids: CAUTION! Concentrated mineral acids are highly corrosive.

7.2.1 Nitric Acid: (HNO_3): Concentrated, trace metals grade or equivalent.

7.2.2 Sulfuric Acid: (H_2SO_4): Concentrated, trace metals grade or equivalent.

7.2.3 Hydrochloric Acid: (HCl): Concentrated, trace metals grade or equivalent.

7.3 Prepared Reagents:

7.3.1 All prepared reagents are labeled accordingly at the time of preparation. This label must include the reagent name, preparation date, the analyst who prepared it, and the expiration date. Expiration dates must conform to the earliest expiration date of any chemical used in the preparation of the reagent. All information pertinent to the prepared reagents must be recorded in a reagent logbook or directly into the LIMS.

7.3.2 5% (wt/wt) Potassium Permanganate Solution (KMnO_4): Prepare by dissolving 100 g of KMnO_4 in 2000 mL of reagent water. This solution has a shelf life of six months. **CAUTION:** strong oxidizer.

7.3.3 5% (wt/wt) Potassium Persulfate Solution ($\text{K}_2\text{S}_2\text{O}_8$): Prepare by dissolving 100 g of $\text{K}_2\text{S}_2\text{O}_8$ in 2000 mL of reagent water. This solution has a shelf life of six months. Method 7470 only.

7.3.4 Sodium Chloride / Hydroxylamine Hydrochloride Solution (NaCl / $\text{NH}_2\text{OH}\cdot\text{HCl}$): (*abbrev. HyHy*); Prepare by dissolving 240 g of NaCl and 240 g of $\text{NH}_2\text{OH}\cdot\text{HCl}$ in 2000 mL of reagent water. This solution has a shelf life of six months.

7.3.5 10% Hydrochloric Acid: (10% concentrated acid by volume.) Prepare by adding 2500 mL of concentrated HCl to a 25L container half-filled with reagent water and bring to the mark with reagent water.

7.3.6 Stannous Chloride Solution (SnCl_2 in HCl): Prepare by dissolving 100 g of SnCl_2 in 10% HCl . Dilute to the 1000 mL mark. The solution has a shelf life of one month. Store in a tightly closed container so that exposure to air is kept to a minimum. This solution should also be kept away from any mercury standard, reagent used in digestion or field sample.

7.3.7 Blank Matrix Solution (BMS): Fill a 2000 mL flask half way with reagent water. Measure 40 mL of concentrated HNO_3 , 80 mL of concentrated H_2SO_4 , 200 mL of KMnO_4 , 80 mL of $\text{K}_2\text{S}_2\text{O}_8$ and 40 mL of Hydroxylamine Hydrochloride. Swirl until solution is clear and colorless. Allow to cool to room temperature and bring to the 2000 mL mark with reagent water.

7.4 Purchased Standards:

- 7.4.1 100 µg/mL Hg Stock Standard #1: 100 ppm Hg#1 (SS). Purchased certified standard -- Certificate to be scanned and the original retained in the Mercury laboratory.
- 7.4.2 100 µg/mL Hg Stock Standard #2: 100 ppm Hg#2 (SS). Purchased certified standard -- Certificate to be scanned and the original retained in the Mercury laboratory. Purchased from a different vendor than #1.
- 7.4.3 Certified Soil Standard: ERA Soil Standard; "Metals in Soil" from *Environmental Resource Associates* – a standard reference material (SRM) containing a certified quantity of Mercury.

7.5 Prepared Standards:

- 7.5.1 All prepared standards must be properly labeled and recorded into a standards logbook or directly into the LIMS. For further information refer to SOP BF-GP-019 "Standards Traceability and Storage".
- 7.5.2 10,000 ng/mL Hg Intermediate Standard #1: 10,000 ppb Hg#1 (IS). Measure 2 mL of concentrated HNO₃ to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 5.0 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased stock standard is expired, whichever comes first.
- 7.5.3 10,000 ng/mL Hg Intermediate Standard #2: 10,000 ppb Hg#2 (IS). Measure 2 mL of concentrated HNO₃ to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 5.0 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased stock standard is expired, whichever comes first.
- 7.5.4 Hg TCLP Spike: Add 5.0 mL of concentrated HNO₃ to a 100 mL Class A volumetric flask half-filled with reagent water. Measure 1.335 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. The final concentration will be 1335 ng/mL. This solution expires in 6 months or when the original purchased stock standard is expired, whichever comes first.
- 7.5.5 100 ng/mL Hg Working Standard #1: 100 ppb Hg#1 (WS). Measure 2.0 mL of concentrated HNO₃ to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 0.5 mL of 10,000 ng/mL Hg Intermediate Standard #1 to the flask and bring to the mark with reagent water. This standard expires 24 hours from the time of preparation and is to be prepared daily.

This standard is to be used for the preparation of the instrument calibration standards.

- 7.5.6** 100 ng/mL Hg Working Standard #2: 100 ppb Hg#2 (WS). Measure 2.0 mL of concentrated HNO₃ to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 0.5 mL of 10,000 ng/mL Hg Intermediate Standard #2 to the flask and bring to the mark with reagent water. This standard expires 24 hours from the time of preparation and is to be prepared daily.

This standard is to be used for the preparation of the initial calibration verification solution (ICV), the continuing calibration verification solution (CCV), the addition of matrix spikes to samples (MS/MSD), and laboratory control samples (LCS).

8.0 Sample Collection, Preservation, Shipment and Storage

- 8.1** Aqueous samples are to be collected in plastic containers and preserved with Nitric Acid to pH < 2. Preserved samples can be stored at room temperature. Sample digestion and analysis must be completed within 28 days of sample collection.
- 8.2** Samples received at the laboratory unpreserved should be kept at 4°C and should be preserved as soon as possible. Allow samples preserved by the laboratory to stand for 24 hours prior to digestion.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	HDPE	50 mL	HNO ₃ , pH < 2;	28 Days	40 CFR Part 136.3
Soils	Glass	3 g	Cool 4 + 2°C	28 Days	N/A

¹Inclusive of digestion and analysis.

9.0 Quality Control

**Refer to the TestAmerica Corporate Quality Assurance Plan for general information and more specific detail. Often project-specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than TestAmerica's QAP or the following criteria. The following criteria are subsequently presented as the minimum criteria of those criteria deemed applicable in the absence of project-specific DQO's.*

9.1 Sample QC

- 9.1.1** Method Blank (MB): A volume of reagent water (method 7470) or measured amount of silicon oxide (method 7471) which is carried through the sample preparation and analysis procedure along with each batch of samples (not to exceed 20 samples). This blank is useful in monitoring for contamination. May also be referred to as a Preparation Blank (PB).
- 9.1.1.1** Aqueous Blank (method 7470/245.1): Add 30 mL of reagent water to a digestion cup. Prepare and analyze as a sample with each batch of samples.
- 9.1.1.2** Soil Blank (method 7471): Add 0.6 g of Silicon (IV) Oxide Oxide (used as a soil matrix substitute) to a digestion cup. Prepare and analyze as a sample with each batch of solid samples.

9.1.1.3 TCLP Blank (method 7470): An associated extraction blank will accompany each set of TCLP extracts (EBLK). Add 30 mL of EBLK to a digestion cup (LB). Prepare and analyze with each batch of extracts. This is in addition to the regular aqueous blank (MB).

9.1.2 Laboratory Control Sample (LCS): A volume of reagent water spiked with a known concentration of mercury, which is carried through the preparation and analysis procedure along with each batch of aqueous samples (not to exceed 20 samples). The LCS is employed to determine method accuracy. May also be referred to as a Laboratory Fortified Blank (LFB).

9.1.2.1 Water LCS (method 7470/245.1): Add 30 mL of reagent water to a digestion cup and fortify with a known amount of mercury (spike with 2.0 mL of 100 ppb Hg#2 = 4 ppb Hg at a final volume of 50 mL). Prepare and analyze with each batch of samples.

9.1.2.2 TCLP LCS (method 7470): Add 200 mL of EBLK to a 500 mL bottle and spike with 1.0 mL of Hg TCLP Spike, and preserve to pH <2 with nitric acid. Add 30 mL of the spiked EBLK to a digestion cup (4 ppb Hg at a final volume of 50 mL). Prepare and analyze with each batch of extracts.

9.1.2.3 Solid LCS (for Wipes or project specific): Add 0.6 g of Silicon (IV) Oxide to a digestion cup and spike with 2.0 mL of 100 ppb Hg#2 (4 ppb Hg at a final volume of 50 mL).

9.1.3 Laboratory Control Sample Standard Reference Material (LCSSRM): Method 7471. A solid matrix material containing a known quantity of mercury, which is carried through the preparation and analysis procedure along with each batch of solid samples (not to exceed 20 samples). The LCSSRM serves the same purpose as the LCS. The quantity of laboratory control sample standard reference material used is selected to give a target result of 4 ppb in a final volume of 50 mL. This amount will vary by manufacturer lot.

Example: A lot of ERA Metals in Soil has a certified mercury concentration of 2.170 mg/kg. To obtain a theoretical instrument result of 4 ppb (µg/L) in a 50 mL final volume, use the following target amount of soil LCSSRM:

$$4 \mu\text{g/L} \times 0.050 \text{ L} \div 2.170 \mu\text{g/g} = 0.09216 \text{ g}$$

9.1.4 Matrix Spikes: For each batch of samples (not to exceed 20 samples), a matrix spike (MS) should be processed on a routine basis. Spiked samples will be used to determine matrix effects on digestion and detection. A representative base sample is selected and a replicate quantity is added to an additional digestion cup. For SW846 and MCAWW this replicate sample is spiked with 2.0 mL of 100 ppb Hg#2 (4 ppb Hg at a final volume of 50 mL).

9.1.5 Duplicates: For each batch of samples (not to exceed 20 samples), replicate samples should be processed on a routine basis. Replicate samples will be used

to determine precision, and are either a method duplicate (DU) or matrix spike duplicate (MS or MSD; typical for SW846 and MCAWW, client assigned for MCP/RCP work). A matrix duplicate is just a replicate preparation of a selected representative base sample. A matrix spike duplicate is just a matrix duplicate that is spiked the same as a matrix spike.

9.1.6 Sample QC frequency and control limits:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Reporting Limit (SW846); < MDL (MCAWW)
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	80-120% recovery (SW846); 85-115% recovery (MCAWW)
Matrix Spike (MS) ²	1 in 20 or fewer (SW846); 1 in 10 or fewer (MCAWW)	75-125% recovery (SW846) 70-130% recovery (MCAWW)
Matrix Spike Duplicate (MSD) ² or Matrix Duplicate (DU) ²	1 in 20 or fewer samples	75-125% recovery (MSD); or RPD < 20% (duplicates)
Laboratory Control Sample Standard Ref. Material (LCSSRM)	1 in 20 or fewer samples	Specified by manufacturer on a per lot basis; typically about 70-130%

¹ Alternately referred to as a Laboratory Fortified Blank (LFB).

² The base sample for MS/MSD is arbitrarily selected, unless specifically requested by a client. MCP/RCP requires if the MS/MSD recovery <30%, the samples will be redigested and reanalyzed. Provide narrative upon confirmation. LCSD is also required for MCP/RCP.

9.2 Instrument QC

9.2.1 All instrument QC standards are prepared and digested daily, typically at the same time as the instrument calibration standards (see section 10.2). Add 25-30 mL reagent water to a digestion cup. Spike with the volume of 100 ppb Hg#2 specified for each standard, and digest for a minimum of 30 minutes. The final QC sample volume is 50 mL.

9.2.2 Initial Calibration Verification (ICV): Prepared as described in 9.2.1 and spiked using 1.5 mL of 100 ppb Hg#2 (3 ppb at a final volume of 50 mL). The ICV checks the accuracy of the calibration and must be the first sample analyzed following a new calibration or at the start of a new analytical sequence.

9.2.3 Initial Calibration Blank (ICB): An unspiked blank sample prepared as described in 9.2.1. The ICB must be analyzed directly after the ICV.

9.2.4 Low Level Calibration Verification (CRA/ICVL/CCVL): Prepared as described in 9.2.1 and spiked using 0.1 mL of 100 ppb Hg#2 (0.2 ppb at a final volume of 50 mL). The CRA is at the same concentration as the lowest non-blank calibration point and is at or near the typical laboratory reporting limit (RL). The CRA/ICVL must be analyzed following the ICV and ICB at the beginning of any an analytical sequence. SW846 Update 4 and many client QAPPs require that an additional CRA/CCVL be analyzed at the end of each analytical sequence.

9.2.5 Continuing Calibration Verification (CCV): Prepared as described in 9.2.1 and spiked with 1.0 mL of 100 ppb Hg#2 (2 ppb at a final volume of 50 mL). The CCV is analyzed at the beginning and end of an analytical sequence, and at a frequency of every 10 samples, ensuring the continued accuracy of the calibration.

9.2.6 Continuing Calibration Blank (CCB): An unspiked blank sample identical to the ICB. The CCB must be analyzed directly after each CCV, and verifies that contamination has not accumulated over the analysis of the previous ten samples.

9.2.7 Serial Dilution (SD): For each sample batch, a representative sample is selected (typically the base sample that is used for the MS/MSD). The sample is diluted 5X (1+4 dilution) using blank matrix solution (BMS) and is analyzed along with the base sample to evaluate sample matrix effects.

9.2.8 Instrument QC frequency and control limits:

Quality Check	Conc.	Frequency	Control Limit
ICV	3.0 ppb	Beginning of each analytical sequence	90-110% recovery (SW846); 95-105% recovery (MCAWW)
ICB	Blank	Beginning of each analytical sequence	< Reporting Limit
CRA/ ICVL/CCVL	0.2 ppb	Beginning of each analytical sequence. End of each analytical sequence for SW846 update 4.	50-150% recovery, 70-130% for MCP/RCP 70%-130% recovery – SW846 Update 4
CCV	2.0 ppb	Every 10 samples	80-120% recovery (SW846); 90-110% recovery (MCAWW)
CCB	Blank	Every 10 samples following each CCV	< Reporting Limit
SD	N/A	1 for each sample batch	+/-10% of base sample

10.0 Procedure

10.1 Sample Preparation

10.1.1 Samples to be prepared are selected from a report of available in-house samples separated by sample matrix and/or analysis method, and sorted by due dates (or any of a variety of user selectable sort criteria). Selected samples are added to a Preparation Bench Sheet (or Batch). For additional detail concerning batch creation criteria and procedures, refer to SOP BF-ME-001.

10.1.2 Samples of different matrix types (e.g. water, solid) or preparation/analysis methods (e.g. 7470, CLP) are typically prepared and analyzed separately. Samples assigned 7470 (SW846) and 245.1 (MCAWW) can be batched and analyzed together; but solid samples are separated from water samples, and CLP samples are separated from non-CLP samples. TCLP extracts may be analyzed with aqueous 7470 samples, but typically are not. Although not a method

requirement, aqueous total samples are batched and analyzed separately from dissolved samples.

10.1.3 Based on the sample batches to be prepared and analyzed together, a sequential numerical order is established for the preparation and digestion cups are labeled as follows: Starting with '1' for the first client sample in the first batch, begin numbering samples sequentially until the base sample for the batch QC (MS/MSD or MD/MS) is reached. Assign the base sample 2 numbers and skip 1 cup leaving the rack position empty. (This is a placeholder for the serial dilution to be created prior to analysis.) The next two cups are the two batch QC samples. Continue with the next client sample and continue to the last client sample, followed by the batch laboratory control sample (LCS) and method blank (BLK). Continue with additional batches of the same type as necessary (up to 88 labeled sample cups – the number of positions on the Leeman autosampler). It is a good practice to clearly mark the cups to be used for spiked batch QC samples (MS/MSD/LCS).

Note: This assigned sequential order is used throughout both the preparation and analysis procedures.

10.1.4 Obtain the appropriate client samples from the cooler(s) or metals sample storage area. Arrange the samples on a sample cart in the order designated above.

10.1.5 Aqueous Sample Digestion (method 7470, 245.1):

10.1.5.1 Making sure the cap is on securely, shake or invert the container several times to homogenize the sample, and pour 30 mL of the sample into the appropriately labeled digestion cup. (Refer to section 5.5 of SOP BF-GP-005 for further instruction on sample homogenization.) Take care to use the appropriate sample bottle when pouring sample, DU/MS or MS/MSD groups. Some clients provide additional bottles for each of the samples in this group, However, much of the time, a single sample bottle will be used for all three aliquots. Reagent Water (30 mL) is used for the LCS and BLK samples. For TCLP extracts, use the pre-spiked MS/MSD and LCS volumes (typically prepared in the Metals Digestion Lab).

10.1.5.2 Spike all MS, MSD, LCS samples as specified in the Batch QC section.

10.1.5.3 Add the following reagents to all samples in each batch:

- 1.0 mL Nitric Acid (HNO_3): **Caution!** Add slowly to leachates. Acid may react vigorously or violently with some samples. Highly reactive samples may require additional nitric acid.
- 2.0 mL Sulfuric Acid (H_2SO_4): **Caution!** Add slowly to leachates. Acid may react vigorously or violently with some samples.
- 5.0 mL Potassium Permanganate (KMnO_4)
- 2.5 mL Potassium Persulfate ($\text{K}_2\text{S}_2\text{O}_8$)

- 10.1.5.4** Cap the digestion cups loosely enough so that pressure does not build up can be evacuated, but also tight enough so that the caps stay on, and that volume loss due to the heating minimized.
- 10.1.5.5** All samples should remain a purple color for at least 15 minutes after adding the potassium permanganate. If any sample becomes clear or otherwise loses its purple color, add an additional 5 mL of the potassium permanganate to ALL samples in the batch, including batch QC samples. If the purple color fades once again, re-prepare the affected sample(s) using a reduced initial volume, noting the volume used on the bench sheet, and dilute to 30 mL using reagent water.
- 10.1.5.6** Put the samples on the hot block (95°C +/- 3°C) for 2 hours. Remove and let cool.
- 10.1.5.7** Uncap each digestion cup and add 2.0. mL of sodium chloride – hydroxylamine hydrochloride (HyHy) to each sample. Allow bubbling to subside, and top each sample to the 50 mL mark with reagent water.
- 10.1.5.8** Replace the cap tightly and shake vigorously for two to three seconds or until most of the purple color has faded. Vent the digestion cup. A brown residue of undissolved manganese dioxide (MnO₂) may remain on the bottom or sides of the digestion cup. It is of no concern, and may dissolve over time.

10.1.6 Solid Sample Digestion:

- 10.1.6.1** Homogenize each sample as described in section 5.6 of SOP BF-GP-005. Add 0.6 g of each sample to the appropriately labeled digestion cup. Take care to use the appropriate sample bottle when weighing sample, DU/MS or MS/MSD groups. Some clients provide additional bottles for each of the samples in this group, However, much of the time, a single sample bottle will be used for all three. For the SRM sample, add the calculated target amount of ERA Standard (see section 9.1.3). For the BLK sample add 0.6g Silicon (IV) Oxide (SiO₂). Add approximately 5 mL reagent water to each cup (enough to cover the sample).
- 10.1.6.2** Spike all MS, MSD, LCS samples as specified in the Batch QC section.
- 10.1.6.3** Add the following reagents to all samples in each batch:
- 1.0 mL Nitric Acid (HNO₃). **Caution!** Acid may react vigorously or violently with some samples. Highly reactive samples may require additional nitric acid.
 - 3.0 mL Hydrochloric Acid (HCl)
- 10.1.6.4** Heat the samples uncapped on the hot block at 95°C (+/- 3°C) for 2 minutes. Remove and let cool.

10.1.6.5 Add the following to all samples in each batch:

- 15 mL Reagent Water
- 15 mL Potassium Permanganate (KMnO_4): **Caution!** KMnO_4 can react with HCl to produce chlorine gas.

10.1.6.6 Cap the digestion cups loosely enough so that pressure does not build up and can be evacuated, but also tight enough so that the caps stay on and that volume loss due to the heating minimized.

10.1.6.7 Put the samples on the hot block at for 30 minutes. Remove and let cool.

10.1.6.8 Uncap each digestion cup and add 6.0 mL of sodium chloride – hydroxylamine hydrochloride (HyHy) to each sample. Allow bubbling to subside, and top each sample to the 50 mL mark with reagent water.

10.1.6.9 Replace the cap tightly and shake vigorously for two to three seconds or until most of the purple color has faded. Vent the digestion cup. A brown residue of undissolved manganese dioxide (MnO_2) may remain on the bottom or sides of the digestion cup. It is of no concern, and may dissolve over time.

10.1.7 Analysis of sample digestates must be performed within 24 hours of digestion or the sample will need to be re-digested.

10.1.8 Some aqueous samples and most solid samples will contain sediments or other solid material that may physically interfere with the analysis by clogging or restricting flow through the instrument sample introduction tubing. These samples may be filtered.

10.2 Calibration

10.2.1 Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. Instrument calibration for mercury analysis is performed at a minimum of each day the analysis is to be performed. A six point linear calibration is used.

10.2.2 Preparation of Calibration Standards: Six standards of known mercury concentration are prepared by dilution of the 100 ppb Hg#1 working standard. Separate calibration curves are digested for water and soils independently, including instrument QC, to match the matrix of the digested samples.

10.2.2.1 Add 20-30 mL Reagent Water to six digestion cups. Spike each cup with the appropriate volume of 100 ppb Hg#1 (see chart below).

10.2.2.2 Digest each standard using the aqueous or soil digestion procedure from section 10.1.5 or 10.1.6 respectively. This ensures that the instrument calibration is matrix matched to the samples to be analyzed. Final volume for the calibration standards is 50 mL.

- 10.2.2.3** Allow the calibration standards to fully cool to room temperature before using (minimum 30 minutes). Failure to allow the standards to cool sufficiently will likely result in the need to recalibrate the instrument.

Calibration Standard	Standard Conc. in 50 mL vol. (ppb)	100 ppb Hg#1 Spike vol. (mL)	Final Conc. Aqueous Samples (µg/L)	Final Conc. Solid Samples (mg/kg)	Control Limit
S1	0	0	0	0	SD < 5000
S2	0.2	0.1	0.33	0.017	%RSD < 30
S3	1.0	0.5	1.67	0.083	%RSD < 5
S4	2.0	1.0	3.33	0.17	%RSD < 5
S5	5.0	2.5	8.33	0.42	%RSD < 5
S6	10.0	5.0	16.67	0.83	%RSD < 5

- 10.2.3** Calibrating the Instrument: This procedure outlines the basic steps to calibrating the instrument. For specific details concerning the operation of the Leeman Analyzer and/or instrument software (WinHg), refer to instrument User's Guide and Manual. For the purposes of this SOP it is assumed that the instrument software and settings are configured for the analysis of samples by the methods covered in this SOP and for the generation of data in a format and manner compatible with TestAmerica Buffalo laboratory operations. It is also assumed the analyst is familiar with and properly trained in the use of the instrument and software.

- 10.2.3.1** Instrument operating parameters have been demonstrated to meet the necessary requirements for the analyses described in this SOP. Instrument parameters may be altered as the need arises, however, significantly altering these parameters may necessitate reevaluation of instrument and method detection limits prior to implementation for sample analysis. Currently used instrument operating parameters are:

- Pump Rate = 7 mL/min
- Gas Flow Rate = 0.7 L/min
- Sample Uptake Time = 10 sec
- Sample Integration Time = 10 sec
- Rinse Time = 40-60 sec

- 10.2.3.2** Perform any needed instrument maintenance prior to calibration. Note any maintenance performed in the instrument maintenance log. Detailed instructions and manufacturer suggested scheduling for the performance of most routine instrument maintenance is available within the instrument software using the "Perform Maintenance" command under the "Utility" menu.

- 10.2.3.3** Turn on Lamp, Pump, and Gas. The lamp will require a minimum of 15 minutes to warm up. For best performance, allow the pump to run 15 minutes to flush the tubing and allow it to settle into place. (For new tubing, allow a 30 minute or longer "break-in" time.)

- 10.2.3.4** Perform a Lamp Adjustment. Record the setting and baseline reading in the maintenance logbook. A large day-to-day change in the lamp setting (without changes to the instrument optical cell or optical bench) may indicate a need for further maintenance.
- 10.2.3.5** Pour the calibration standards to the appropriate cups on the autosampler. The 6 standards and concentrations are currently set up in the S1-S6 positions.
- 10.2.3.6** Use 'StdAuto' to analyze 3 replicates of each of the 6 calibration standards (select S1-S6 and Repetitions 1-3) to generate the calibration curve. In addition to the control limits specified above, a correlation coefficient (ρ) of at least 0.995 is required; however, for best performance it is strongly recommended that the correlation coefficient be greater than 0.9995. A lower value may indicate a need for instrument maintenance or poorly prepared calibration standards.
- 10.2.3.7** Calibrations are evaluated in accordance with SOP BF-GP-006, and against the criteria specified in this section. If all criteria are met, accept the calibration. Print a copy of the calibration screen and export the calibration to a data file. Attach a copy of the calibration screen to each data set analyzed using that calibration.

10.3 Sample Analysis

- 10.3.1** Pre-Run Setup Checklist: Sample analysis is only performed on a properly maintained and calibrated instrument. Prior to beginning an analytical sequence verify or perform the following steps. Note: these items are not necessarily in a specific sequential order.
 - 10.3.1.1** Select analyst initials in the "User Name" on the "Main" tab:
 - 10.3.1.2** Lamp is on and warmed up (at least 15 min); Gas is turned on; Pump is on. Controls for all 3 are on the "Control" tab.
 - 10.3.1.3** Instrument is calibrated and calibration is accepted. "Cal Curve" tab.
 - 10.3.1.4** Stannous Chloride bottle has sufficient volume, and 10% HCl rinse carboy has sufficient volume.
 - 10.3.1.5** Calibration Check Standard cups (Instrument QC) have sufficient volume. Current setup has instrument QC in the following autosampler rack positions: C1 = ICB/CCB; C2 = ICV; C3 = CCV; C4 = CRA/CCVL.
 - 10.3.1.6** Create a data file from the "File" menu, or select an existing file from the "Dataset" field. The file contains information which designates the instrument used, date and sample analysis/batch matrix type using the following convention:

- A single letter instrument identifier (Leeman2 = H; Leeman 3 = J).
- Numeric date: month, day (2 digits each), and year (1 digit); e.g. Jan 15, 2008 = 01159.
- A two character analysis/batch matrix type identifier
 - C# = Calibration data file
 - D# = Dissolved Hg
 - S# = Total Hg - Solids
 - TC = TCLP extracts
 - W# = Total Hg – Waters

Example: The second analytical sequence (run data file) of aqueous samples for total Hg analyzed on August 12th, 2009 using the Leeman 2 instrument would have a Data File name of H08129W2.

10.3.1.7 For the samples to be analyzed, label test tubes using the same numbering scheme used for the digestion cups.

10.3.1.8 Pour approximately 8-10 mL of each sample from the digestion cups into the test tubes, and place them in a 44 position autosampler rack.

10.3.1.9 Create the serial dilution (SD) samples: Combine 2 mL of the sample to be diluted with 8 mL of Blank Matrix Solution (BMS). The dilution factor for the SD sample is 5 (@5 in the autosampler table).

10.3.1.10 Create Autosampler files (using the Rack Editor) containing all of the batch samples and calibration check standards (instrument QC) to be analyzed. Autosampler table files are named similar to Data Files substituting #1, #2, #3, etc. in place of the 2-character sample type identifier. One file is needed for each rack of up to 44 samples. Autosampler table columns are populated as follows:

- cup#: This column is pre-populated from 1-44 (the number of positions per sample rack). The cup number should match up with the digestion cup number and test tube number for the 1st rack of 44 samples. For the 2nd rack (if needed), the cup# will equal the digestion cup number minus 44.
- sample ID: Batch sample IDs (up to 10 characters) may be typed (or scanned from a barcode) into this column..
- extended ID: Batch sample IDs (up to 20 characters) are typed (or scanned from a barcode) into this column. If sample ID is longer than 20 characters, the 3 digit lab code may be removed to provide more space.
- weight and volume: these columns are not used and are pre-populated with 1.0000

- ? A D F P S U SC UI US... (Cup Macro Column): This column uses macro codes to send instructions to the instrument software. All instrument QC samples are analyzed by including the check standard cup position (C1=ICB/CCB; C2=ICV; C3=CCV; C4=CRA/CCVL). The CP macro code tells the instrument to execute the preceding macro codes prior to analyzing the sample in that cup #; otherwise, the macro codes in a given row execute following analysis of the sample in that cup.
- Macro Code Layout: The following macro codes are used for a typical analysis (see example table layout below):
 - Cup#1 = C2 C1 C4 C3 C1 CP
 - Cup#10,20,30, etc = C3 C1
 - .
 - Last Cup = C3, C1 or C3, C1, C4 as required)

10.3.1.11 Select the Autosampler file(s) and corresponding cup positions to be analyzed on the "Sample" Tab. Check that the samples/sample racks are in their proper positions on the autosampler.

10.3.1.12 Select "Run Auto" on the "Sample" tab. A full rack of 88 cups including all QC will take about 3.0 - 3.5 hours.

10.3.1.13 Samples with results outside of the calibration range must be diluted to within range and reanalyzed. If sample dilutions are required then add them to the end of the run. Append the autosampler table as needed with the sample ID. The dilution factor is added to the 'extended ID' column preceded by the '@' character. Include any check standard macro codes as appropriate.

10.3.1.14 Perform a preliminary on-screen review of the data for QC failures or other requirement compliances (eg QAPPs). Due to the 24h holding time constraint in analyzing mercury digestates, it is strongly recommended that the analyst perform any needed reanalysis (not requiring sample redigestion) immediately and within the same data file. This will reduce the need for unnecessary sample redigestion and simplify data review and reporting.

Example Autosampler Table for Batch 48552.

cup	sample ID	extended ID	weight	volume	? A D F P S U SC UI C1..C7
1		mb 48048552/1-a	1.0000	1.0000	C2 C1 C4 C3 C1 CP
2		lcs 48048552/2-a	1.0000	1.0000	
3		480-15035-d-1-c	1.0000	1.0000	
4		480-15035-d-2-c	1.0000	1.0000	
5		480-15035-d-3-c	1.0000	1.0000	

cup	sample ID	extended ID	weight	volume	? A D F P S U SC UI C1..C7
6		480-15035-d-4-c	1.0000	1.0000	
7		480-15035-d-5-c	1.0000	1.0000	
8		480-15035-d-6-c	1.0000	1.0000	
9		480-15035-d-7-c	1.0000	1.0000	
10		480-15035-d-8-c	1.0000	1.0000	C3 C1
11		480-15035-d-9-c	1.0000	1.0000	
12		480-15035-d-10-c	1.0000	1.0000	
13		480-15035-d-11-c	1.0000	1.0000	C3 C1 C4
14			1.0000	1.0000	

The above table would result in a run sequence as follows:

```

ICV
ICB
CRA/ICVL
CCV
CCB
↑
10 samples (with the given ID#s)
↓
CCV
CCB
↑
3 samples
↓
CRA/CCVL
CCV
CCB

```

10.3.2 Post-Analysis Checklist: Performed once all sample analyses are complete.

10.3.2.1 In the WinHg Database program 'Report' tab, select the appropriate data file to be reported. (Samples to be reported can be selected using a combination of the 'Batch List' and 'Records List' sections.) Select 'Generate Report'. Reports can be generated on-screen ("Report" Format, 'Viewer' Destination -- viewable via the 'Viewer' tab), to the printer ('Report' Format, 'Printer' Destination), or to a file ('PRN File' Format, "Disk File" Destination). The current report format setting is 'HgRpt'.

10.3.2.2 Generate a printed report of the raw data, and a PRN disk file (used to import data to the LIMS). The file should be named the same as the instrument data file that the samples were run in. Save the file directly to the H-Drive (Lab Data) in the folder *H:\Mercury\Lims*. Click 'Generate' after creating the file to write the data to the file. Raw

instrument files are backed up to a network drive in accordance with SOP BF-IS-010.

10.3.2.3 Record solution ID#s for the ICV, ICB, CRA/CCVL, CCV, and CCB directly onto the raw data report.

10.3.2.4 Attach a hard copy (screen-shot) of the Calibration Curve screen to the printed raw data report. Record the following information on the Calibration page:

- Analysis Date
- Analyst Initials
- Instrument Name
- Solution ID#s for the Calibration Standards
- Calibration File Name (eg H08129C1)
- Data File Name the calibration was used for (eg H08129CW)
- Batch ID#s for the analyzed batches

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV / CRA / LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS / MSD \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.3 Wet-Weight Basis

$$\text{Sample Concentration (mg/kg)} = C \times V/W$$

Where: C = concentration in extract (mg/L)

V = Volume of the digestate (L, 50 mL = 0.05 L)

W = Weight of sample aliquot (not dried) extracted (g × 0.001 = kg)

11.4 Percent Solids

To report percent solids in solid samples, calculate as follows:

$$\% \text{ Solid (S)} = DW / WW \times 100$$

Where: DW = Sample weight (g) dried (dry weight)

WW = Sample weight (g) before drying (wet weight)

11.5 Dry-Weight Basis

Sample Concentration (mg/kg) = $(C \times V) / (W \times S)$, or

Sample Concentration (mg/kg) = $C \times V / WW$

11.6 Calculation of extract concentrations are automatically done by the system's software.

11.7 Calculation of sample concentrations from measured extract concentration are done by the LIMS system.

11.8 Contingencies for Handling Out-of Control or Unacceptable Data

11.8.1 Data is to be evaluated in accordance with SOPs BF-GP-012 and BF-ME-013.

11.8.2 If an ICV, ICB, or opening CRA/ICVL falls out of acceptance limits, discontinue the analysis to correct the problem, and then restart the analysis. Note: Instrument re-calibration may be required.

11.8.3 If any CCV or CCB falls out of acceptance limits, the preceding and following 10 samples must be evaluated. If a LCS or BLK fails, the entire batch of samples must be evaluated.

- For high CCVs and LCSs, non-detect samples may be accepted. All other affected samples must be reanalyzed.
- For low CCVs, all affected samples must be reanalyzed. For low LCSs, the batch must be re-prepared and reanalyzed.
- For High CCBs and BLKs, non-detect samples may be accepted. Samples greater than 10x the CCB or BLK result may also be accepted. All other affected samples need to be reanalyzed or re-prepared and reanalyzed.

11.8.4 A Job Exception Report form or Non-Conformance Memo (NCM) may need to be filed if extensive problems are noted within any one sample or analysis. The analyst performing the run completes these forms. A Job Exception Report form should be completed and filed with the Project Manager and QA Manager for any of the following conditions:

- Holding times exceeded
- Insufficient sample volume for re-digestion
- Re-digestion required due to sample batch QC failure
- Unusual sample matrix or sample reactivity which requires deviation from this SOP.

11.8.5 In the event of unknown positives or sample matrix which presents the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action.

11.8.6 When an out of control situation occurs, the analyst must use his/her best judgment and use any available resources to determine the corrective action to be taken. The analyst may need to seek immediate assistance from the supervisor, laboratory director, project manager, QA personnel or other experienced members of the staff if he/she is uncertain of the proper course of action. The test may need to be stopped until the problem is corrected since the problem may be instrumental and not chemical. Out of control data will never be released without the approval of the Supervisor, QA Manager, or Laboratory Director.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL SOP BF-QA-001. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed. MDLs are verified on each instrument to which they will apply via annual MDLV studies. Instrument detection limits (IDL) are determined for each instrument on a quarterly basis.

12.2 Training Requirements

12.2.1 Analyst training will adhere to requirements specified in SOP BF-QA-004

12.2.2 The department supervisor has the responsibility to ensure that this procedure is performed by analysts with the required experience and properly trained in its use.

12.2.3 The analyst must complete laboratory safety orientation training that includes, but is not limited to, PPE requirements, chemical handling, and electrical safety.

12.2.4 The analyst must read the SDS for all chemicals used in this method.

12.2.5 The analyst must read and understand the contents of this SOP and the Method used as a reference for this SOP.

12.2.6 The analyst must successfully complete a Demonstration of Capability (DOC) before training in this method is deemed to be complete.

12.3 Demonstration of Capability (DOC)

12.3.1 Initial Demonstration of Capability is performed upon completion all other aspects of training. A completed IDOC is the final step of analyst training and allows the analyst to perform the method without trainer supervision.

12.3.2 Continuing Demonstration of Capability is performed annually. This ensures that the analyst has remained proficient in performing the method and no retraining is necessary.

12.3.3 DOC will be performed as described in SOP BF-QA-004 section 5.8.

13.0 Pollution Control

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

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention".

14.2 The following waste streams are produced when this method is carried out:

- Acidic waste from samples and sample digests. Waste generated will contain Nitric Acid and will therefore be disposed of as "AN" waste in accordance with SOP BF-WM-001.

15.0 References / Cross-References

15.1 EPA Method 7470A – Mercury in Liquid Waste

15.2 EPA Method 7471A – Mercury in Solid Waste

15.3 EPA Method 7471B – Mercury in Solid Waste

15.4 40 CFR Part 136 (MCAWW) (Revision B), "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act" U.S. Environmental Protection Agency.

15.5 EPA 600/4-79-020 Methods 245.1, Revision B; SW-846, 3rd Edition, Method 7470A

15.6 "Method 1631, Revision B: Mercury in Water by Oxidation" (40 CFR 136, Revision B) U.S. Environmental Protection Agency, May 1999.

15.7 The following SOPs have been referenced, or are relevant to, procedures described in this document, and should be referred to for more detailed information on the indicated topics:

- BF-ME-001 Metals Department Batching Procedure
- BF-ME-013 Metals Data Review
- BF-GP-001 Autopipets (Eppendorfs), Syringes, Repipettor Calibration
- BF-GP-002 Balances, Reagent Water, Temperature Control Devices
- BF-GP-003 Glassware Cleaning
- BF-GP-004 Dry Weights
- BF-GP-005 Sample homogenization and sub-sampling
- BF-GP-006 Initial Calibration Evaluation
- BF-GP-011 Sample Storage and Handling
- BF-GP-012 Data Review Requirements
- BF-GP-019 Standard Storage and Traceability
- BF-GP-020 Thermometer Calibration
- BF-IS-010 Instrument Data File Backup
- BF-WM-001 Waste Management
- BF-QA-001 Determination of MDLs
- BF-QA-004 Personnel Training (for DOC's)

16.0 Method Modifications:

Item	Method xx	Modification
01	7470/7471/ 245.1	The volumes have been minimized for preparation of all methods listed, although the chemistry remains unchanged. This change fits our preparation equipment and minimizes waste.
02	7470/7471/ 245.1	<i>Environmental Express</i> Hot Blocks and plastic digestion cups replace Hot Plates and BOD bottles for sample preparation.

17.0 Attachments

17.1 Attachment 1: Manufacturer recommended positioning of the computer/analyzer/ autosampler system.

17.2 Attachment 2: Sample Water Digestion Batch Bench Sheet

17.3 Attachment 3: Sample Soil Digestion Batch Bench Sheet

17.4 Attachment 4: Example Instrument Calibration Page

18.0 Revision History

- Revision 9, dated October 1, 2015
 - o Section 9.1.2.2: Changed volume of EBLK used to 200 mls.
 - o Section 9.1.2.2: Changed spike volume used to 1.0 ml.
 - o Lab Director Change, signature added.
- Revision 8, dated September 23, 2014
 - o Approval Section updated, signatures added
 - o Throughout document: changed CRA to either CRA/ICVL or CRA/CCVL to include nomenclature change for SW846 Update 4 methods.
 - o Section 10.1.5 Changed preparation reagent volumes to better match relative quantities in reference methods. Potassium persulfate changed from 2mL to 2.5mL. Hydroxylamine hydrochloride solution changed from 2.5mL to 2mL.
 - o Section 10.1.6 Changed preparation reagent volumes to better match relative quantities in reference methods. Potassium permanganate changed from 10-15mL to 15mL. Hydroxylamine hydrochloride solution changed from 2.5mL to 6mL. Changed the two additions of reagent water from 5-10mL each to 5mL and 15mL.
- Revision 7, dated April 25, 2013
 - o Section 6.2.2 added reference to include both beginning and ending temperature measurements.
 - o Section 10.3.1.10 changed sample ID to include use for sample ID's less than 10 characters.
 - o Section 10.3.1.10 removed reference to S and P macro Code combination for determination of spike recoveries (no longer used).
 - o Section 12.1 removed reference to yearly MDL determination
 - o Section 9.1.5 addition of MCP/RCP assignment of MS/MSD
 - o Section 9.1.6 added MCP/RCP to LCSD and MS/MSD criteria
 - o Table 9.2.8 added CRA criteria of 70-130% for MCP/RCP
 - o Quality Manager updated, signature added

- Revision 6, dated March 28, 2012
 - o Section 10.2.2 added reference to include soil digestion to match the matrix of the digested samples.
 - o Section 10.2.2.2 addition of 10.1.6 to reference the soil digestion procedure.
- Revision 5, dated January 19, 2012
 - o Changed Standard Reference Material (SRM) to Laboratory Control Sample Standard Reference Material (LCSSRM). Throughout.
 - o Changed LCV to CRA throughout.
 - o Changed Method Blank (BLK) to Method Blank (MB) throughout.
 - o Changed Matrix Duplicate (MD) to Matrix Duplicate (DU) throughout.
 - o Changed Serial Dilution (SRD) to Serial Dilution (SD).
 - o 9.1.1.3 Changed TCLP extraction blank TALS ID from BLK to LB.
 - o 6.2.7 Changed scale model from Mettler AE200 to Denver P-214.
 - o 10.2.3.3, 10.3.1.4 removed references to the rinse bath.
 - o 10.3.1.10 Due to character limitations batch sample IDs are entered into extended ID instead of sample ID.
 - o Updated Example Autosampler Table to reflect changes in section 10.3.1.10.
 - o Deleted S P out of Example Autosampler Table.
 - o 10.3.2.2 H-Drive folder changed from Ward/Sdgs/Instdata/Mercury to H:\Mercury\Lims.
- Revision 4, dated 15 February 2011
 - o Replaces previous SOP BF-ME-011, revision 3
 - o Added reference to Method 7471B in Section 1.2 and Section 15.0
 - o Replaced all references to Blank Spikes (BS) to Laboratory Control Samples (LCS).
 - o Added hot block temperature range of +/-3°C to Section 6.2, 10.1.5 and 10.1.6.
- Revision 3, dated 25 January 2010
 - o Replaces previous SOP BF-ME-011, revision 2
 - o Spelling, Grammar, & Formatting corrections
 - o Section 4.1.9 Added specific guideline for sample reanalysis to check for carryover from high level samples
 - o Section 4.2 & 4.3 Additional detail provided concerning various chemical vs. physical interferences
 - o Section 9.1.2.3 Removed references to AFCEE and USACE
 - o Sections 6.26, 6.27, 6.2.10, 10.2.3.7 Added missing cross-references to other relevant SOPs
 - o Attachment 4 - Title renamed for clarity

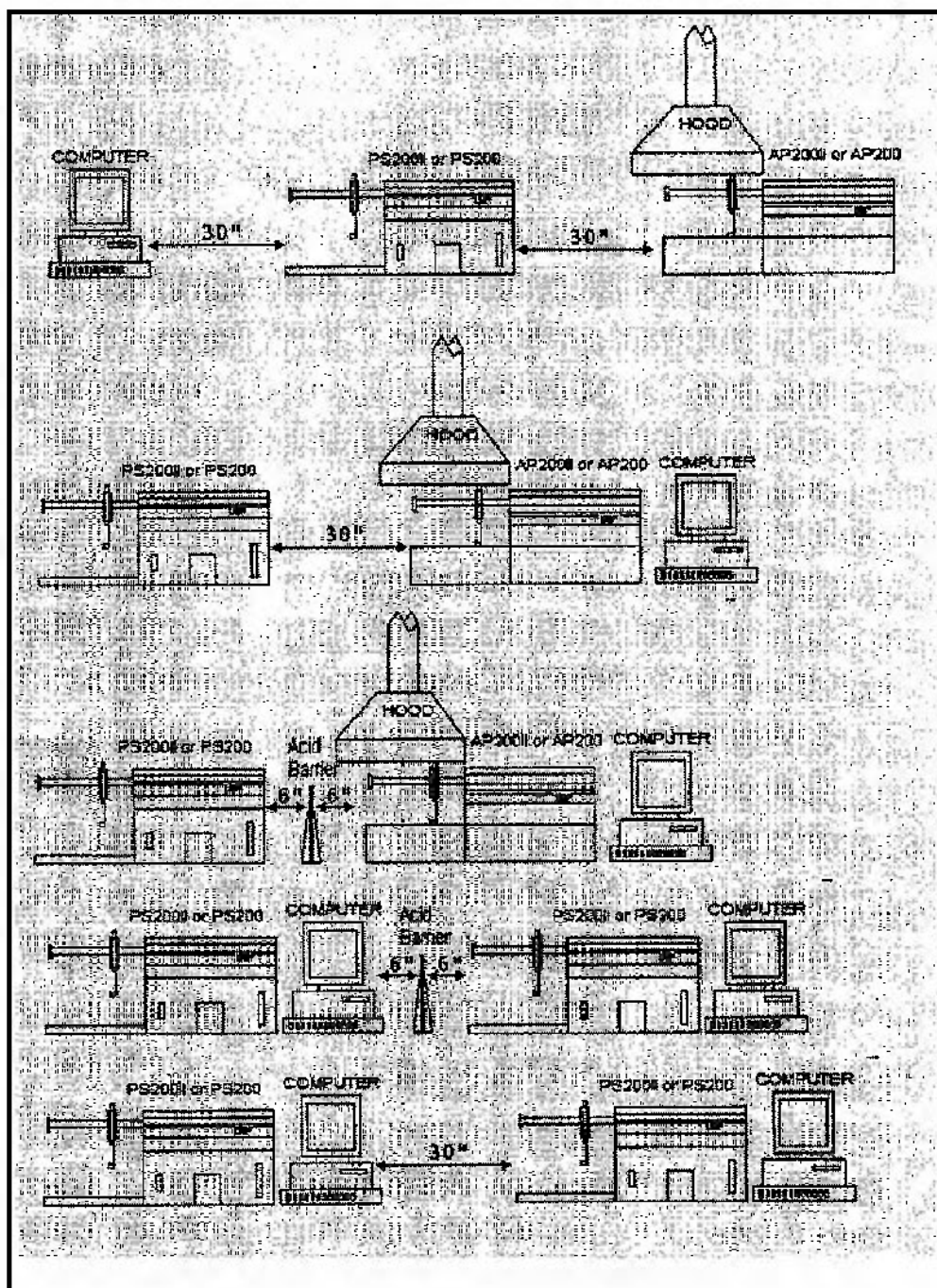
- Revision 2, dated 02 September 2009
 - o Replaces previous SOP BF-ME-011, revision 1
 - o Section format changes from STL to TestAmerica Standard format completed. Several section numbers have changed, and several new tables have been included. References to outdated SOP reference numbers updated.
 - o Incorporated contents of interim change from July 07, 2008 concerning concentration of purchased mercury stock standards (1000 ppm → 100 ppm) and concentrations of prepared intermediate standards (20,000 ppb → 10,000 ppb, and elimination of 2000 ppb).
 - o Changed spike amount for the blank spike for aqueous total mercury batches from 2.0 ppb to 4.0 ppb so that the blank spike and matrix spike levels are now the same, which is in better accordance with SW-846. The 2.0 ppb blank spike had been implemented to accommodate AFCEE/USACE. A 2.0 ppb blank spike will still be used for AFCEE/USACE at which point that becomes necessary.
 - o Corrected Table 9.1.6 Sample QC control limits to agree with EPA methods:
 1. MS/MSD %recoveries from 20% to 25% (SW846) and 30% (MCAWW)
 2. MD/MSD %RSD from 30% to 20%
 - o Changes to several sections reflecting differences in operation between AIMS and ELEMENT LIMS systems; including the following abbreviation changes:
 1. CRA → LCV
 2. SD → MSD
 3. LCS and LFB → BS or SRM
 4. MBLK → BLK
 - o Added details concerning data file and autosampler file naming conventions
 - o Added details concerning sample preparation cup and analysis test tube numbering system
 - o Added an example autosampler table for demonstration of use of extended ID field for sample IDs longer than 10 characters, and for improved clarity in demonstrating the use of macro codes for execution of instrument QC.
 - o Reformatted sample preparation section to separate Aqueous from Solid digestion steps
 - o Rewrote calibration and sample analysis sections to better depict current use of instrument software in setting up, calibrating, and sample analysis. Added section on instrument operating parameters
 - o Added Sample Calibration Page Attachment
 - o Updated Example batch attachments from AIMS to ELEMENT batches
 - o Changed potassium permanganate added to soil samples from 10 mL to 10-15mL.

- Revision 1, dated 15 April 2008
 - o Replaces previous SOP BF-ME-011, revision 0
 - o Sections 10.3.7, 10.3.8, 10.3.9 and 10.4. Edited for improved clarity regarding stock standard, intermediate standard, and working standard preparation; and to correct copying errors introduced in transitioning SOP formats from AME-MERCURY-50, rev.7 to BF-ME-011, rev.0.
 - o Section 12.7 and 12.8. Correct 100 ppb to Hg#2 instead of Hg#1
 - o Sections 10.4.3 and 10.4.4. Moved reference to preparation of MS/SD, LCS, and LFB from Section 10.4.3 to Section 10.4.4.
 - o Section 12.1.2. Changed quantity of water added to soil MBLK to 10 mLs and removed "Carry the MBLK through the entire digestion process" (redundant with 12.1)
 - o Section 14.4. Added addition of approximately 10 mLs reagent water to cover soil samples

- Revision 0, dated 30 November 2007
 - o Replaces previous SOP AME-MERCURY-50, revision 7
 - o Section 9.2.5 correct to weekly to daily for pipette verification
 - o Sections 12.2.2, 12.2.3, 12.3, and 12.6 correct 100ppb to Hg#2 instead of Hg#1
 - o Section 12.4 correct from 1:3 to 1:5 serial dilution
 - o Section 14.26 replace 40CFR with MCAWW
 - o Section 14.31 deleted turn off argon gas valve

Attachment 1

Manufacturer recommended positioning of the computer/analyzer/autosampler setup.



Attachment 2

Sample Water Digestion Batch Bench Sheet (Page 1 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-5412

Analyst: Kaczalski, Jason

Batch Open: 2/14/2011 9:25:00AM

Method Code: 480-7470A_Prep-480

Batch End: 2/14/2011 11:25:00AM

Preparation, Mercury

	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments	Output Sample Lab ID
1	MB-480-4850/17-A N/A	N/A		30 mL	50 mL	N/A	N/A	N/A		
2	LCS-480-4850/18-A N/A	N/A		30 mL	50 mL	N/A	N/A	N/A		
3	480-1514-D-1 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
4	480-1514-D-1-SD (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
5	480-1514-D-1-MS (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
6	480-1514-D-1-MSD (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
7	480-1514-D-2 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
8	480-1514-D-3 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
9	480-1514-D-4 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
10	480-1518-G-1 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
11	480-1518-G-2 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
12	480-1518-G-3 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
13	480-1569-F-1 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		
14	480-1569-F-2 (7470A)	N/A	Filtrate	30 mL	50 mL	2/15/11	10_Days - R	2		

Printed: 2/14/2011

Page 1 of 5

TestAmerica Buffalo

Attachment 2

Sample Water Digestion Batch Bench Sheet (Page 2 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

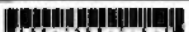
Batch Number: 480-5412

Analyst: Kacalski, Jason

Batch Open: 2/14/2011 9:25:00AM

Method Code: 480-7470A_Prep-480

Batch End: 2/14/2011 11:25:00AM

15	480-1568-F-3 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2	
16	480-1568-F-4 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2	
17	480-1568-F-5 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2	
18	N/A	N/A				N/A	N/A	N/A	
19	N/A	N/A				N/A	N/A	N/A	
20	N/A	N/A				N/A	N/A	N/A	

Attachment 2

Sample Water Digestion Batch Bench Sheet (Page 3 of 5)

Metals/Inorganics Analysis Sheet (To Accompany Samples to Instruments)		
Batch Number: 480-5412 Method Code: 480-7470A_Prep-480	Analyst: Kacalski, Jason	Batch Open: 2/14/2011 9:25:00AM Batch End: 2/14/2011 11:25:00AM
Batch Notes		
Uncorrected Temperature	95.0	
Oven, Bath or Block Temperature 1	95.0	
Uncorrected Temperature 2		
Oven, Bath or Block Temperature 2		
Digestion Tube/Cup Lot #	1010192-0328	
Hood ID or number		
Hot Block ID number	A	
ID number of the thermometer	A-02-24-10	
Temperature		
Lot # of Nitric Acid	-RT12894-	
Lot # of hydrochloric acid		
Sulfuric Acid Lot Number	RT13092	
Potassium Permanganate Lot Number	027468	
Potassium Persulfate Lot Number	20419	
Hydroxylamine Sulfate Lot Number		
Stannous Chloride Lot Number	038576	
Hydroxylamine Hydrochloride Lot	039590	
NaCL Lot #		
Repletor Volume Check		
SOP Number		
Printed : 2/14/2011 Page 3 of 5 TestAmerica Buffalo		

Attachment 2

Sample Water Digestion Batch Bench Sheet (Page 4 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-5412

Analyst: Kacalski, Jason

Batch Open: 2/14/2011 9:25:00AM

Method Code: 480-7470A_Prep-480

Batch End: 2/14/2011 11:25:00AM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 480-4850/18-A	MEH_HG2_WKG_00002	2.0 mL	50 mL		
480-1514-D-1 MS	MEH_HG2_WKG_00002	2.0 mL	50 mL		
480-1514-D-1 MSD	MEH_HG2_WKG_00002	2.0 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

Attachment 3

Sample Soil Digestion Batch Bench Sheet (Page 1 of 4)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-5415

Analyst: Kacalski, Jason

Batch Open: 2/14/2011 10:30:00AM

Method Code: 480-7471A_Prep-480

Batch End: 2/14/2011 11:00:00AM

Preparation, Mercury

	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments	Output Sample Lab ID
1	MB-480-5415/1 N/A	N/A		+0.0250 g	50 mL	N/A	N/A	N/A		
2	LCSSRM-480-5415/2 N/A	N/A		+0.0677 g	50 mL	N/A	N/A	N/A		
3	480-1409-B.5 (7471A)	1342	Solid	+0.0609 g	50 mL	2/11/11	8_Days - R	4		
4	480-1553-C-1 (7471A)	N/A	Solid	+0.0374 g	50 mL	2/16/11	8_Days - R	2		
5	480-1553-C-1-SD (7471A)	N/A	Solid					2		
6	480-1553-C-1-M5 (7471A)	N/A	Solid					2		
7	480-1553-C-1-MSD (7471A)	N/A	Solid	+0.0230 g	50 mL	2/16/11	8_Days - R	2		
8	480-1553-B-2 (7471A)	N/A	Solid	+0.0139 g	50 mL	2/16/11	8_Days - R	2		
9	N/A	N/A				N/A	N/A	N/A		
10	N/A	N/A				N/A	N/A	N/A		

Attachment 3

Sample Soil Digestion Batch Bench Sheet (Page 2 of 4)

Metals/Inorganics Analysis Sheet (To Accompany Samples to Instruments)		
Batch Number: 480-5415 Method Code: 480-7471A_Prep-480	Analyst: Kacalski, Jason	Batch Open: 2/14/2011 10:30:00AM Batch End: 2/14/2011 11:00:00AM
Batch Notes		
Acid used for pH adjustment _____		
Perform Calculation (0=No, 1=Yes) _____		
Nominal Amount Used _____		
SOP Number _____		
Digestion Tube/Cup Lot # 1010192-0328 _____		
Hot Block ID number B _____		
Hood ID or number _____		
Balance ID 25850472 _____		
Blank Soil Lot Number RT05542 _____		
Lot # of Nitric Acid -RT12894- _____		
ID number of the thermometer A-02-24-10 _____		
Lot # of hydrochloric acid 026990 _____		
Potassium Permanganate Lot 027468 _____		
Sulfuric Acid Lot Number _____		
Hydroxylamine Sulfate Lot Number _____		
Potassium Persulfate Lot Number _____		
Stannous Chloride Lot Number 039576 _____		
Uncorrected Temperature 95.0 _____		
Oven, Bath or Block Temperature 1 95.0 _____		
Uncorrected Temperature 2 _____		
Printed : 2/14/2011 Page 2 of 4 TestAmerica Buffalo		

Metals/Inorganics Analysis Sheet (To Accompany Samples to Instruments)														
Batch Number: 480-5415	Analyst: Kacalski, Jason	Batch Open: 2/14/2011 10:30:00AM												
Method Code: 480-7471A_Prep-480		Batch End: 2/14/2011 11:00:00AM												
<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 30%; padding: 2px;">Oven, Bath or Block Temperature 2</td> <td style="padding: 2px;"></td> </tr> <tr> <td style="padding: 2px;">NaCl Lot #</td> <td style="padding: 2px;"></td> </tr> <tr> <td style="padding: 2px;">Reptitator Volume Check</td> <td style="padding: 2px;"></td> </tr> <tr> <td style="padding: 2px;">Aqua Regia Lot Number</td> <td style="padding: 2px;"></td> </tr> <tr> <td style="padding: 2px;">Hydroxylamine Hydrochloride Lot</td> <td style="padding: 2px;">039590</td> </tr> <tr> <td style="padding: 2px;">Batch Comment</td> <td style="padding: 2px;">Eppie: HGL-5</td> </tr> </table>			Oven, Bath or Block Temperature 2		NaCl Lot #		Reptitator Volume Check		Aqua Regia Lot Number		Hydroxylamine Hydrochloride Lot	039590	Batch Comment	Eppie: HGL-5
Oven, Bath or Block Temperature 2														
NaCl Lot #														
Reptitator Volume Check														
Aqua Regia Lot Number														
Hydroxylamine Hydrochloride Lot	039590													
Batch Comment	Eppie: HGL-5													
Comments														

Sample Soil Digestion Batch Bench Sheet (Page 4 of 4)

TestAmerica Buffalo

Attachment 4

Example Instrument Calibration Page

SPM2E CALIBRATION PAGE

WinHq Database 1.1

File Utility Help

Protocol: **hgppb** Dataset/Proto: **H08219W1/hgppb**

Protocol Line Info Cal Curve Report Ctrl Chart Viewer

Reset

Calb Coeffs

New Cal

Update Coeffs

Spike Coeffs

A: B: **1.74758e-5** C: **-2.24870e-2**

rho: **696677** Type: **Linear**

Rel Abs: **572164** Accepted

Accept New

Include **S8** Rep 1 ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐

S	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3
01	0.0000	-0.024	-0.024	.94	1548	-1875	661	934
02	20.0000	.186	-0.015	11881	8.22%	11580	12971	11083
03	1.0000	1.02	.018	59526	3.07%	60717	60444	57420
04	2.0000	2.00	.002	115863	0.36%	115786	116304	115496
05	5.0000	5.04	.042	289819	0.92%	292874	288723	287863
06	10.0000	9.98	-.023	572164	0.27%	572662	573419	570412

Conc. 10.0

Ready CAP NUM

8-21-2009
L2 (M)

9-21-09-K
-11
-12
-13
-14
-15

9061710
-11
-12
-13
-14
-15

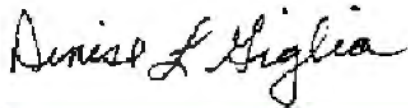
Cal: H08219C1
Run: H08219W1
Batch: 9H20039
9H20040
9H20041

Data Review: 8/10/09 **For**
2nd Review: 8/24/09 **LPH**

**Title: Analytical Methods for the Analysis of GC/MS Volatiles
[SW-846 Method 8260C]**

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):



Denise Giglia
Department Manager

1/13/15

Date



Kenneth Kasperek
EHS Manager

1/13/15

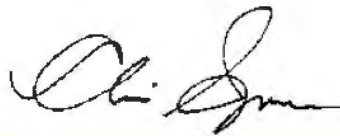
Date



Brad Prinzi
Quality Assurance Manager

1/13/15

Date



Christopher Spencer
Laboratory Director

1/13/15

Date

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods 8260C -5 mL aqueous purge, 8260C – 5gr soil and 8260C – medium level soil.

1.1.2 Applicable matrices include all aqueous samples, sediment, and soil.

1.1.3 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve. For a 5-ml purge volume, the RL for the majority of compounds is 1 ug/l.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1 This analytical method is utilized for the analysis of water, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1.

2.2 The method includes sample preparation and analyses by purge and trap gas chromatograph/mass spectrometer (GC/MS). Method can be used for 5mL purge.

2.3 Volatile compounds are extracted from sample matrix by the purge and trap method. Analytes are desorbed onto a capillary column. An appropriate ramping temperature program is applied to maximize separation and achieve the correct resolution between the analytes. A mass spectrometer detector (MSD) interfaced to the gas chromatograph (GC) is utilized to detect analytes of interest.

2.4 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

3.0 Definitions

3.1 MB - Volatile blank: MB's are made from laboratory produced volatile free water. They are analyzed before samples to ensure a clean laboratory environment and analytical system.

3.2 IBLK – Instrument Blank: IBLK's are made from laboratory produced volatile free water. They are analyzed after high level samples to verify that the system is clean and demonstrate the absence of carryover.

3.3 LCS – Laboratory Control Sample: An LCS consists of a sample of volatile free water that is spiked with a group of target compounds representative of the method analytes. It is used to monitor the accuracy of the analytical process, independent of matrix effects.

- 3.4 Surrogates (System Monitoring Compounds):** Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, MB, LCS and MS/MSD are spiked with surrogates.
- 3.5 MS/MSD – Matrix Spike/Matrix Spike Duplicate:** A Matrix Spike is an environmental sample which is spiked with a group of target compounds representative of the method analytes. A Matrix Spike Duplicate is a second aliquot of the same sample, which is spiked with the same target compounds. These samples are used to evaluate accuracy and precision in environmental samples.
- 3.6 Batch:** A batch is a set of 18 field samples using the same procedures within the same time period. Using this method each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.
- 4.0 Interferences**
- 4.1** Airborne contamination may result from solvent vapors. MBs and IBLKs will be utilized to demonstrate a clean system and laboratory environment.
- 4.2** Some volatile compounds can permeate through a sample septum seal during storage or shipment. A weekly volatile holding blank is stored in all sample incubators to monitor contamination.
- 4.3** Contamination by carryover can occur whenever a sample with high concentrations of target compounds precedes a sample with low levels. The purging device, syringe and lines are flushed between every analysis to reduce carry over contamination. The trap is baked between each analysis.
- 5.0 Safety**

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

5.1 Specific Safety Concerns or Requirements

5.1.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap devices

- Varian Archon Auto sampler
- Encon Concentrator
- O/I Analytical Auto sampler and Concentrator
- Centurion Auto sampler

6.1.2 Trap Packing

- Vocarb 3000
 - o Carbpac B
 - o Carboxen 1000
 - o Carboxen 1001
- OI #10
 - o Tenax
 - o Silica Gel
 - o cms
- Other traps may be used if the Quality Control criteria are met.

6.1.3 Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP5890, MS:

- Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624
- Internal diameter: 0.25mm or 0.18mm
- Length: 20m, 30m or 60m.
- Coating: Cyanopropylphenyl Methyl Silicone
- Film thickness: 1.0um or 3.0um

6.1.4 Data System

- Computer with Chemstation enviroquant software
- Gas Chromatograph/Mass Spectrometer (GC/MS)-GC: HP6890 or HP7890, MS: Hewlett-Packard/Agilent 5973N or 5975.
- ProLab Resources software

6.1.5 Analytical Balance Mettler - Toledo Inc. Mettler AE160**6.2 Supplies**

- Syringes - Hamilton Syringes size, 10ul, 25ul, 50ul, 100ul, 500ul, 1ml, 5ml, 10ml, 25ml
- Pasteur Pipettes – disposable
- Vials and caps - 2ml disposable
- Vials and caps - 40ml disposable
- Volumetric flasks - Pyrex 2ml, Pyrex 10ml, Pyrex 50ml, Pyrex 100ml
- pH paper - wide range -.EM Science

7.0 Reagents and Standards

7.1 Reagent Water - For volatile analysis, the reagent water is volatile free and is prepared by passing water through a carbon trap.

7.2 Methanol - Burdick & Jackson, purge and trap grade

7.3 Stock Standards - Are purchased as certified standard mixtures. Traceability is documented following the procedures in the "Standards Traceability and Preparation Logbooks" SOP# BF-GP-019. Individual compounds are prepared using reagent grade chemicals following the "Primary Standards Preparation" SOP# BF-MV-010.

7.3.1 Stock Target Compound Mix – Is composed of two different mixtures.

7.3.1.1 Gas Mix (See Table 8 for component list) is purchased at a concentration of 2000ug/ml.

7.3.1.2 8260 Mega Mix (See Table 9 and 10 for component list) is purchased and is composed of five separate mixtures.

- 8260 List 1/ Standard 1 is purchased at a concentration of 2000ug/ml.
- 8260 List 1/Standard 2 is purchased at a concentration of 10000ug/ml.
- 8260 List 1/ Standard 4 is purchased at a concentration of 2000ug/ml.
- 8260 List 1/Standard 5 is purchased at a concentration of 20000ug/ml.

- 8260 List 1/Standard 6 is purchased at a concentration of 4000ug/ml.

7.3.1.3 8260 Add Mix (See Table 13 for component list) is purchased and is composed of six separate mixtures.

- 8260 List 2/ Standard 1 is purchased at a concentration of 2000ug/ml.
- 8260 List 2/Standard 2 is purchased at a concentration of 2000ug/ml.
- 8260 List 2/ Standard 3 is purchased at a concentration of 20000ug/ml.
- 8260 List 3/Standard 1 is purchased at a concentration of 20000ug/ml.
- Buffalo Add Mix A is purchased at a concentration of 5000ug/ml.
- Buffalo Add Mix B is purchased at a concentration of 1000ug/ml.

7.3.2 Stock Calibration Verification Mix – Is composed of two different mixtures.

7.3.2.1 Second Source Gas Mix (See Table 11 for component list of 8260 second source standards) is purchased at a concentration of 2000ug/ml.

7.3.2.2 Second Source 8260 Mega Mix is purchased and is composed of five separate mixtures.

- Second source 8260 List 1/ Standard 1 is purchased at a concentration of 2000ug/ml.
- Second source 8260 List 1/Standard 2 is purchased at a concentration of 10000ug/ml.
- Second source 8260 List 1/ Standard 4 is purchased at a concentration of 2000ug/ml.
- Second source 8260 List 1/Standard 5 is purchased at a concentration of 20000ug/ml.
- Second source 8260 List 1/Standard 6 is purchased at a concentration of 4000ug/ml.

7.3.3 Stock Internal Standard Solution – A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and Fluorobenzene in Methanol is purchased at a concentration of 2500ug/ml.

7.3.4 Stock System Monitoring Solution – A mixture of Toluene-D8, 4-Bromofluorobenzene, Dibromofluoromethane and 1,2-Dichloroethane-d4 in Methanol is purchased at a concentration of 2500ug/ml.

7.3.5 Stock BFB Solution – A solution of 4-Bromofluorobenzene in Methanol is at a concentration of 25000ug/ml.

7.4 Working Standards

7.4.1 Intermediate Calibration Solution (Two individual mixtures)

7.4.1.1 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up to a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.4.1.2 1000ul of each of the five stock standard 8260 Mega Mixes are added to approximately 5ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol.

7.4.2 A Full List Matrix Spike Standard is made from stock Calibration Standards and is composed of two mixes.

7.4.2.1 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.4.2.2 1000ul of each of the five stock standard 8260 Mega Mixes are added to approximately 5ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol.

7.4.3 Working Internal Standard and System Monitoring Compound Solutions – for auto injection by instrument.

7.4.3.1 Working Internal Standard Solution - An Internal Standard Mixture is made from IS stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.

7.4.3.2 Working System Monitoring Calibration Solution - A System Monitoring Compounds Mixture is made from Surrogate stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.

7.4.4 Tuning Mixture - 4ul of stock solution 4-Bomofluorobenzene (BFB) tuning mixture is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

7.4.5 Working Initial Calibration Standards

7.4.5.1 Water: 5 mL (7 point curve)

7.4.5.1.1 50ul, 25ul, 12.5ul, and 5ul each of Intermediate Calibration Solution (7.4.1) is added to reagent water in each of four 50ml volumetric flasks for multi-point surrogate calibration. The flasks are brought to volume with reagent water to prepare the 100, 50, 25 and 10 ug/L standards respectively. Single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.4.5.1.2 2.5ul, 1ul and 0.5ul of each Intermediate Calibration Solution (7.4.1) are added to reagent water in each of the 100ml volumetric flask. The flask is brought to volume with reagent water to prepare the 5ug/L, 1ug/L and the 0.5ug/L standard.

7.4.5.1.3 The standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.4.5.2 Soil: (7 point curve)

7.4.5.2.1 100ul, 50ul, 25ul, and 10ul each of Intermediate Calibration Solution (7.4.1) is added to reagent water in each of four 50ml volumetric flasks for multi-point calibration. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, and 20 ug/kg, standards respectively. Single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.4.5.2.2 10ul, 5ul and 2.5ul of each Intermediate Calibration Solution (7.4.1) are added to reagent water in each of the 100ml volumetric flask. The flask is brought to volume with reagent water to prepare the 10, 5.0 and 2.5 ug/Kg standards respectively.

7.4.5.2.3 5 ml of each standard is then transferred into seven individual 40ml vials and loaded onto the auto sampler.

7.4.6 Continuing Calibration Standard

7.4.6.1 Water: 5 ml

7.4.6.1.1 12.5ul of stock target compound mix is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 25ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.4.6.2 Soil:

7.4.6.2.1 25ul of stock target compound is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 50ppb. Take 5ml and transfer it into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.5 Storage of Standards

7.5.1 Stock standards are stored in flame sealed ampoules at 22⁰ C to –20⁰ C according to the vendor's specifications.

7.5.2 Secondary dilution standards are stored in Teflon-sealed crimp cap vials at < 0⁰ C.

7.5.3 Aqueous standards are stored in Teflon-sealed vials at 4⁰ C $\pm$ 2⁰ C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are collected in 40 mL vials with caps and septa, preserved to a pH < 2 with Hydrochloric Acid and stored at 4 $\pm$ 2 degrees C until time of analysis.

8.2 Holding time for unpreserved samples is 7 days from sample date. For preserved samples the holding time is 14 days from sample date.

8.3 For some clients, regulatory agencies or QAPPS, the specified holding times may be different than those described in 8.2. In those cases, consult the specific Protocol/Method/QAPP or Project Manager for holding time details.

8.4 **Sample Storage**

- Volatile samples are stored at $4 \pm 2^{\circ}\text{C}$ from the time of collection until analysis.
- Volatile samples are stored together in refrigerators specifically designated for volatiles only.
- Storage blanks are stored with samples until analysis.
- Samples and extracts are stored separately.
- Volatile samples and standards are stored separately.

8.5 **Preparation Of MS/MSD Samples**

8.5.1 Water Samples: 40ml vial is spiked 11ul of 100ng/ul for the 5ml purge. This corresponds to a final concentration in the samples of 25ug/L. Analysis proceeds according to procedures described for water analysis.

8.5.2 Low Level Soil/Sediment Samples: 2.5ul of 100ng/ul of matrix spiking solution is added to a 5g aliquot of sample. This corresponds to a final concentration in the samples of 50 ug/kg. Analysis proceeds according to procedures described for low-level soil/sediment samples.

8.5.3 Medium Level Soil/Sediment Samples: 1ml of methanol containing the soil spiking solution is combined with 50 mL of water. Sample analysis proceeds according to procedures described for medium level soil/sediment samples.

9.0 **Quality Control**

9.1 **Blank Analysis**

9.1.1 Method Blank: A method blank consisting of a clean reference matrix (reagent water or purified quartz sand) must be analyzed prior to the analysis of samples but following any standard analysis.

- Target compounds detected in a method blank must fall below the reporting limit, unless specified in client QAPP.
- If internal standard or systems monitoring compound recoveries are not met, the method blank must be reanalyzed before the analysis of samples.

9.1.2 Storage (Holding) Blank: A weekly holding blank is analyzed to determine if cross contamination occurs within the volatile holding area. The results are reviewed by the quality assurance department and deemed acceptable or not acceptable. Corrective action, if necessary, will be taken.

9.1.3 Instrument Blank: An instrument blank consisting of a clean reference matrix analyzed after the analysis of samples containing target compounds which exceed the calibration range. Multiple instrument blanks are shot until the instrument blank meets the criteria for method blanks.

9.2 Matrix Spike Blank (LCS) An aliquot of clean reference material spiked with the matrix spiking solution is analyzed with each analytical batch.

9.2.1 If a compliant Second Source Calibration Verification (ICV) has already been analyzed, then standards from the primary (CCV) source may be used. The solution is spiked at a concentration of 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.

9.2.2 Alternatively, a standard that is purchased from an alternate vender (or where not available from a second vendor an alternate lot will be used) from the continuing (CCV) standard may be used. The solution is spiked at a concentration of 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.

9.2.3 The LCS must fall within internally derived statistical control limits or where applicable the limits specified by a project QAPP.

9.2.4 Analytes that have been identified as a Poor Performing Compounds (Table 5) will be considered compliant as long as their percent recovery exceeds 10%.

9.2.5 The LCS a 'full-compound' spike will be prepared and the LCS will be spiked with all compounds of interest. Due to the potentially large number of target compounds for method 8260C, it is possible that a few of the spiking compound could fall outside limits in the LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable.

9.2.6 If the results of sample matrix spikes fall outside of the quality control range due to matrix, the MSB is used to verify that the laboratory can perform a spike on a clean matrix.

9.3 Matrix Spike And Matrix Spike Duplicate Analysis A matrix spike and matrix spike duplicate consisting of an actual field sample which has been spiked with the matrix spiking solution.

9.3.1 Matrix spike and matrix spike duplicate analysis will not be performed on rinsates or field/trip blanks.

9.3.2 If a sample has not been designated for MS/MSD analysis by the client, a sample will be selected at the analyst's discretion. MS/MSD analysis will be performed at a minimum of every 20 samples.

9.3.3 If insufficient sample was received for a designated MS/MSD the client will be contacted with the laboratories in-house designated sample for MS/MSD analysis. If no MS/MSD is required, the instance will be documented in the SDG narrative.

9.3.4 If medium level analysis is required on the client designated sample, the laboratory analyst will choose a low level sample on which to perform the quality control analysis. Medium level QC will also be performed.

9.4 Data Assessment & Acceptance Criteria for QC Measures

9.4.1 Technical Acceptance Criteria For Initial Calibration

9.4.1.1 Minimum Response Factors

9.4.1.2 See Table 6 in this SOP for the 8260C method specific minimum response factors. If the % RSD of any of the target analytes should be 20% or less, the average response factor is assumed constant and the average response factor may be used for quantitation. Due to the large number of compounds, some compounds will fail to meet these criteria; any samples with positive detection under this calibration must be flagged as estimated. If more than 10% of the compounds fail to meet the criteria a new calibration is required. Alternate Quant ions maybe used per instrument per initial calibration to maximize RRF factors.

OR

If the % RSD of a target analyte is greater than 20%, linear regression or quadratic regression may be used providing the coefficient of determination is greater than or equal to 0.99. If quadratic regression is used, a minimum of 6 calibration points must to be analyzed.

9.4.1.3 Non-standard analytes are sometimes requested for analysis by this method. For these analytes it is acceptable to analyze a single point standard at the reporting limit with each continuing calibration rather than a five point calibration. If the analyte is not detected in the associated samples a non-detect will be reported and no further action is required. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.

9.4.1.4 For linear curve fit analytes in the initial calibration a read back concentration of the low level calibration standard is evaluated. The read back concentration for linear fit analytes should be 30% of the true value.

9.4.1.5 Second Source Calibration Verification The initial calibration should be verified by analysis of a standard from a second source immediately following the calibration. This is also referred to as an ICV.

9.4.1.5.1 Following the analysis of an acceptable initial calibration curve, an aliquot of this independent standard is analyzed at the CCV level.

9.4.1.5.2 Recoveries of all compounds shall fall within $\pm 30\%$ of the expected values or up to 50% for our defined poor performing compounds listed in Table 5a.

9.4.2 Technical Acceptance Criteria For Continuing Calibration

9.4.2.1 Minimum Response Factors - An initial calibration check or CCV is made daily or during every 12 hour analytical shift. Each compound must meet its minimum response factor (see Initial Calibration Criteria). If the minimum response factors are not met, then the system should be evaluated and corrective actions must be taken prior to sample analysis.

9.4.2.2 Percent Difference - Used to check the validity of the initial calibration. The % Difference for each compound shall be less than or equal to 20% from the initial calibration for the continuing calibration to be valid. Due to the large number of compounds in a calibration, some compounds may fail this criterion. If more than 20% of the compounds fail to meet this criterion a new calibration is required. For any sample detections for an analyte that fails high in the CCV every effort will be made to reanalyze the samples under a compliant CCV. If re-analysis is not possible the analytes will be flagged as estimated and noted in the case narrative. Any sample non-detects for an analyte that fails low in the CCV criterion must have an MDLV standard analyzed to demonstrate sensitivity to the reporting limit. The criterion for the RL standard is that the analyte be detected only. If an analyte is found in the poor performers list in Table 5a fails the 20% criterion no corrective action will be taken until the %D is greater than 50%.

9.4.2.3 Internal Standard Retention Time – The retention times for all internal standards must be evaluated to make sure that they are no more than 10 seconds from that of the midpoint of the initial calibration. If the retention time shift is greater than 10 seconds, the system must be inspected for malfunctions and maintenance must be performed, as required.

9.4.2.4 Internal Standard Response – The EICP area for all internal standards must be evaluated to make sure that they have not change by a factor greater than two (-50% to +100%) from that of the midpoint of the initial calibration. If the response exceeds these limits, the system must be inspected for malfunctions and maintenance must be performed, as required.

9.4.3 Technical Acceptance Criteria of Quality Control Samples

Samples, blanks, matrix spikes, and matrix spike duplicates must meet internal standard and system monitoring compound recovery limits. Where the Internal Standard recovery limit equals sample internal standard characteristic ion area (EICP) divided by the CCV internal standard characteristic ion area (EICP), multiplied by 100.

9.5 Corrective Action for Out-of-Control Data

9.5.1 Corrective Actions For MS/MSD

- 9.5.1.1** If the recoveries of the internal standards and system monitoring compounds do not agree with the unspiked sample (i.e. the sample recoveries were within control limits and MS/MSD recoveries were outside of control limits) the MS/MSD will be evaluated. The analyst will use their technical judgment to determine if the non-conformance is due to sample matrix or laboratory error. If it is determined that the QC failure was due to laboratory error, then reanalysis will occur.
- 9.5.1.2** If the recoveries of the internal standards and system monitoring compounds agree with the unspiked sample (i.e. both the sample and MS/MSD recoveries were outside of control limits) re-analysis is not required. The instance will be documented in the SDG narrative.
- 9.5.1.3** The laboratory on an annual basis establishes limits for the matrix spiking compounds. If the concentrations determined in the MS/MSD do not meet the control limits, no corrective action is necessary as long as the LCS was within control limits. The instance will be documented in the job narrative.

9.5.2 Corrective Actions For Initial Calibration

- 9.5.2.1** If technical acceptance criteria cannot be met, it may be necessary to re-analyze the initial calibration. If after re-analysis, the criteria have not been met, it may be necessary to inspect the GC/MS system for possible problems.
- 9.5.2.2** Corrective actions may require one or several of the following procedures:
- Open new/remake standard mixes
 - The ion source may be cleaned
 - The column may be cut at the injection port end
 - Change the purge trap on the purge and trap unit
 - Correct purge gas flow to optimize response
 - The column may be baked out
 - The purge trap may be baked out
 - The column may be replaced

9.5.3 Corrective Actions for Failure to Meet the Continuing Calibration Acceptance Criteria

- 9.5.3.1** If the technical acceptance criteria given above are not met, it may be necessary to re-analyze the continuing calibration check. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.
- 9.5.3.2** A single point standard at the reporting limit may be analyzed before the analysis of any samples. If the analyte is not detected in the associated samples a non-detect will be reported and a comment in the case narrative will be made. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.
- 9.5.3.3** Other Corrective actions may be taken. The following details possible corrective actions:
- Open new/remake standard mixes
 - The ion source may be cleaned
 - The column may be cut at the injection port end
 - The trap on the purge and trap unit may be replaced
 - The purge gas flow may be adjusted
 - The column may be baked out
 - The trap may be baked out
 - The column may be replaced

9.5.4 Corrective Actions For Samples

- 9.5.4.1** If the internal standard or system monitoring criteria are not met, the sample must be re-analyzed to insure that it was not an internal problem that affected recoveries. If, after re-analysis, recoveries are outside of control limits, a matrix effect can be assumed.
- 9.5.4.2** When dilutions are performed, target compound concentration must fall within the upper range of the initial calibration. If any target compound exceeds the calibration range, the sample would require dilution. The sample immediately following a sample with target compounds above the calibration range must be monitored to insure that there is no carryover present. If there is a possibility of carryover, that sample must be re-analyzed.

- 9.5.4.3** If matrix effects exist, and both analyses exhibit recoveries outside of control limits, both analyses will be reported and documented in the job narrative.
- 9.5.4.4** If, after re-analysis, recovery criteria are met, only the second analyses will be reported. If the second analyses occur outside of the contract required holding time, both analyses will be reported in that instance.
- 9.5.4.5** In the case of a matrix spike or matrix spike duplicate, these samples should only be reanalyzed if an error was identified in preparation or analysis of the sample. Failures will be documented in the SDG narrative.

9.5.5 Corrective Actions for Failure to Meet the Laboratory Control Sample (Matrix Spike Blank) Acceptance Criteria

- 9.5.5.1** The laboratory on an annual basis establishes limits for the matrix spiking compounds. The LCS must fall within these control limits. The LCS will be spiked with 8260 Mega Mix compounds. Due to the potentially large number of target compounds for method 8260C, it is possible that a few of the spiking compounds could fall outside limits in the LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable.
- 9.5.5.2** If the technical acceptance criteria are not met, it may be necessary to re-analyze the matrix spike blank. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.
- 9.5.5.3** Other Corrective actions may be taken. The following details possible corrective actions:
- Open new/remake standard mixes
 - The ion source may be cleaned
 - The column may be cut at the injection port end
 - The trap on the purge and trap unit may be replaced
 - The purge gas flow may be adjusted
 - The column may be baked out
 - The trap may be baked out
 - The column may be replaced

9.5.6 Corrective Actions for Failure to Meet the Method Blank (MB) Acceptance Criteria

- 9.5.6.1** If the technical acceptance criteria are not met, it may be necessary to re-analyze the associated samples.
- 9.5.6.2** If the analyte is a common laboratory contaminant (Methylene Chloride, Acetone, 2-Butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.
- 9.5.6.3** If the target analyte is not greater than the reporting limit in the samples with the non-compliant blank, the data may be reported with the analyte qualified.
- 9.5.6.4** If surrogate recoveries are not acceptable, the data may be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination.

9.5.7 Contingencies for Handling Out-of-Control or Unacceptable Data

- Inform project manager for client input and fill out job exception report.
- Rerun samples to confirm results.
- Resample if client or project manager requests.

10.0 Procedure**10.1 Calibration & Standardization****10.1.1 Instrument Tuning and Performance Check:**

The GC/MS system is calibrated using Perfluorotributylamine (PFTBA) according to the recommended tuning conditions suggested by the vendor.

An instrument performance check of Bromofluorobenzene (BFB) is analyzed at the beginning of each 12-hour analysis period.

The analysis of the instrument performance check is performed using the following procedure:

- 1ul of a 50ng/ul solution is directly injected, resulting in a 50ng injection of BFB into the GC/MS.
- A blank containing 50 ng BFB is purged.

10.1.2 The mass spectrum of BFB is acquired using the following procedure:

- The apex scan, one scan immediately preceding the apex and one scan immediately following the apex are averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB.
- A scan across the peak at one half the peak height may be averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak.
- A single scan of the peak may also be used for the evaluation of the tune. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak
- The mass spectrum of BFB must pass the technical acceptance criteria given in Table 2.

10.1.3 Initial Calibration (ICAL):

The instrument performance check must meet the technical acceptance criteria prior to the analysis of an initial curve or samples. The GC/MS system is calibrated using a minimum of five levels of concentrations. All compounds of interest are included. (See section 9.4 for initial calibration acceptance criteria.)

Solutions containing target compounds and system monitoring compounds are analyzed at the following concentrations:

5 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/L)
VSTD0005	MeOH	100ng/ul	0.5	100	0.5
VSTD001	MeOH	100ng/ul	1	100	1
VSTD005	MeOH	100ng/ul	5	100	5
VSTD010	MeOH	100ng/ul	5	50	10
VSTD025	MeOH	100ng/ul	12.5	50	25
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100

5 gram (soil) Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/kg)
VSTD00025	MEOH	100ng/ul	2.5	100	2.5
VSTD005	MeOH	100ng/ul	5.0	100	5
VSTD010	MeOH	100ng/ul	10	100	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100
VSTD200	MeOH	100ng/ul	100	50	200

10.1.4 Continuing Calibration Verification (CCV):

Every 12 hours of sample analysis the laboratory must demonstrate that the instrument has drifted or changed minimally by performing an instrument performance check and continuing calibration verification. (See section 9.4 for continuing calibration acceptance criteria.)

10.2 Before Analysis

10.2.1 Once initial calibration criteria has been met, and prior to analyzing samples and required blanks, Each GC/MS system must be routinely checked by analyzing a Continuing Calibration Verification (CCV) standard containing all compounds (including internal standards and system monitoring compounds) at a concentration of 25ug/L for 5ml analysis or 50ug/Kg for soil.

10.2.2 If time remains after initial calibration criteria have been met, it may not be necessary to perform a CCV. The 25 ug/L (50ug/Kg for soil) standard may be evaluated against the new initial curve and used as the CCV.

10.2.3 If there is no time remaining in the 12-hour period, the instrument performance check (BFB) must be analyzed along with a new CCV.

10.2.4 Procedure for Continuing Calibration:

10.2.4.1 5ml Water: 12.5ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.

10.2.4.2 Soil: 25ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is transferred to a sample vial. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis

10.3 Sample Analysis

10.3.1 BFB tuning criteria and GC/MS calibration verification must be met before sample analysis begins.

- 10.3.2** The acquisition time of the BFB tune establishes a 12hr. batch. The CCV, MSB, and MB must be analyzed within 12hrs, unless specified by the client request. The remaining time in the 12hr batch is utilized to run samples of similar matrix. The time of initiation of purging is considered the injection time. All aqueous samples are considered a water matrix. All solid samples, with the exception of sludges, are considered soil matrix. Sludges are run medium level.
- 10.3.3** Samples and standard solutions are brought to ambient temperature before analysis.
- 10.3.4** Prior to the analysis of samples, a method blank must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for method blanks must be met prior to sample analysis.
- 10.3.5** Within the analytical batch an LCS must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for the LCS must be met with each batch.

10.4 Water Sample Analysis

- 10.4.1** A 5ml sample aliquot is spiked with internal and system monitoring compounds to a final concentration of 25 ug/L each. The spike may be performed manually with a Hamilton gas tight syringe or the auto sampler may be used. The sample is then loaded onto the auto sampler where it is in turn transferred to the purge chamber.
- 10.4.2** The sample is purged for 11.0 ± 1 minute at ambient temperature.
- 10.4.3** At the end of the purge time, the sample is desorbed onto the gas chromatograph column by rapidly heating the trap from 190°C to 250°C (depending on manufacturer specifications) while the trap is back flushed with Helium between 20 - 60 ml/minute according to the manufactures specifications. The sample is desorbed onto the column and the gas chromatograph temperature ramping program is initiated.
- 10.4.4** While the trap is in the bake mode, the purge chamber is flushed with two 5ml aliquots of reagent water in order to avoid possible contamination from carryover of target compounds.
- 10.4.5** After the sample has desorbed, the trap is conditioned from 190°C to 260°C according to the manufactures specifications. After baking, the trap is ready for the next sample.
- 10.4.6** Dilutions may be necessary if the concentration of any target compound exceeds the working range of the calibration.

10.4.7 In the event that a dilution is required, a measured volume of sample is added to a volumetric flask then brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described.

10.5 Low Level Soil/Sediment Sample Analysis

10.5.1 The bulk low level soil method is based on a heated purge of a 5g sample mixed with reagent water containing a final concentration of 50 ug/L of internal and system monitoring compounds.

10.5.2 For the bulk soil, if a dilution of the soil/sediment is required, a smaller portion of soil may be used. The smallest amount of soil that may be used is 0.5g. If a higher dilution is required, the sample must be analyzed as a medium level soil/sediment.

10.5.3 Initial and continuing calibrations that are used for the quantitation of low soils/sediments are analyzed using the same purge and trap conditions as samples.

10.5.4 Internal standards and system monitoring compounds are added to the sample immediately prior to heating and purging by the auto sampler.

10.5.5 After reagent water is added, the soil/sediment sample is heated to $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$ then purged for 11 ± 1 minutes.

10.5.6 For the EnCore™ sampling devices, at least three per sampling point should be received and require preparation within 48hrs of sampling. Two of the sampling devices are removed and immediately placed into 40mL vials. The weight is taken and directly uploaded into the preparation batch in TALs for GCMS. To these vials 5mL of volatile free water is added and the vials are capped. The soil from the third Encore device is removed and immediately placed into a 20mL vial. The sample weight is then directly uploaded into the preparation batch in TALs for GCMS. The soil is then spiked with the appropriate surrogate standard. 10mL of Purge and Trap grade Methanol for Mass Spectroscopy is added to the vial and the cap is applied. The vial is then shaken for 2 minutes. The vials are then stored at $\geq -7^{\circ}\text{C}$ in an incubator specifically for 5035 volatile samples.

10.5.7 The Terracore™ sampling process is a kit of 3 pre-weighed vials, 2 with 5mL water and 1 with 5mL methanol, are sent out to the field. The samplers use the soil sampler provided to add approximately 5 grams of soil to each of the 3 vials. The vials are then shipped back to the lab and frozen within 48 hours of sampling. Prior to analysis the samples are thawed and brought to room temperature. The weight is taken and directly uploaded into the preparation batch in TALs for GCMS.

10.5.8 The soil/sediment sample is heated $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$ then purged for 11 ± 1 minutes.

- 10.5.9** After purging, the sample is subjected to desorbing as described for water analysis.

10.6 Medium Level Soil/Sediment Samples

- 10.6.1** The medium level bulk soil/sediment method is based on an extraction of a 5gram sample with methanol.
- 10.6.2** 1ml of system monitor compound mixture is then added to the sample.
- 10.6.3** A 9ml aliquot of methanol is quickly added to the sample, bringing the final volume to 10ml. The vial is capped and the sample are either left in methanol for 1 day or sonicated @ 40 deg for 30 minutes prior to analysis.
- 10.6.4** A pre-determined amount of the methanol extract is added to a 50ml volumetric flask, brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described in section 10.4.
- 10.6.5** The soil from the third Encore™ device is removed and immediately placed into a 20mL vial. The sample weight is then directly uploaded into the preparation batch in TALs for GCMS. The soil is then spiked with the 1mL of surrogate standard. 9mL of Purge and Trap grade Methanol for Mass Spectroscopy is added to the vial and the cap is applied. The vial is then shaken for 2 minutes. The vials are then stored at $\geq -7^{\circ}\text{C}$ in an incubator specifically for 5035 volatile samples.
- 10.6.6** If sample extracts are prepared in the field (e.g. Terracore kits) then both system monitoring compounds and internal standards are added by the auto-sampler prior to analysis.
- 10.6.7** Table 3 may be used to determine the volume of methanol extract required for a given dilution factor.

10.7 pH Determinations For Water Samples

- 10.7.1** After the sample aliquots are taken from the VOA vials, the pH of the sample is determined using wide range pH paper. A checkmark will be entered in the injection logbook if the sample pH is <2 , however if the sample demonstrates a $\text{pH} > 2$, the actual pH will be noted in the injection logbook.

11.0 Calculations / Data Reduction

11.1 Calculations For MS/MSD Samples

- 11.1.1** The calculations to determine concentrations are the same equations described for sample analysis of a given matrix.

11.1.2 The percent recovery of the matrix spiking compounds is determined using equation:

$$\text{Matrix Spike Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

Where: SSR = Spiked sample result
SR = Sample results
SA = Spike added

11.1.3 The relative percent difference (RPD) of the recoveries of each compound between the matrix spike and matrix spike duplicate is determined using equation:

$$\text{RPD} = \frac{|\text{MSR} - \text{MSDR}|}{\frac{1}{2} (\text{MSR} + \text{MSDR})} \times 100$$

Where: MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

11.2 Calculations For Initial Calibration

11.2.1 The relative response factor (RRF) for each target compound and each system monitoring compound is calculated using equation.

$$\text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Table 4)

C_{is} = Concentration of the internal standard

C_x = Concentration of the compound to be measured

11.2.2 The relative response factor of the Xylenes requires the use of the area response and the concentration of the peak that represents the single isomer.

11.2.3 The relative response factor of 1,2-dichloroethene is calculated using the sum of the areas of both isomers and the sum of the concentrations.

11.2.4 The average response factor (RRF) is calculated for all compounds of interest.

11.2.5 The relative standard deviation (% RSD) is calculated over the working range of the curve for all compounds using equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}$$

Where,

X_i = each individual value used to calculate the mean

$\bar{X}$ = the mean of n values

n = the total number of values

11.3 Calculations For Continuing Calibration

11.3.1 The relative response factor (RRF) for all target compounds and system monitoring compounds is calculated using equation 11.2.1.

11.3.2 The percent difference between the initial calibration and the continuing calibration is determined for all target compounds and system monitoring compound using equation:

$$\%Difference = \frac{RRF_c - RRF_i}{RRF_i} \times 100$$

Where,

RRF_c = Relative response factor from continuing calibration standard

RRF_i = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

11.4 Percent Moisture Determinations

11.4.1 Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tarred weigh pan. The sample is then dried at 105°C. The sample is allowed to cool. The final weight is recorded. Using the equation for % moisture, concentrations relative to the dry weight of the soil/sediment samples, may be determined.

$$\%moisture = \frac{\text{g of wet sample} - \text{g of dry sample}}{\text{g of wet sample}} \times 100$$

11.5 Quantitation of volatile target compounds is done using the internal standard method. The Internal Standard RRF of the continuing calibration is used in the quantitation calculation.

11.5.1 Water Samples: The following equation is used to calculate water samples:

$$\text{Concentration ug/L} = \frac{(Ax) (Is) (Df)}{(Ais) (RRF) (Vo)}$$

Where,

Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

Ais = Area of the characteristic ion (EICP) for the specific internal standard (see Table 4)

Is = Amount of internal standard added in nanograms (ng)

RRF= Relative response factor from the ambient temperature purge of the calibration standard.

Vo = Volume of water purged in milliliters (mL)

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5. If no dilution is performed, Df = 1.

11.5.2 Low Level Soil/Sediment Samples - The following equation is used for low level soil/sediment samples:

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax) (Is)}{(Ais) (RRF) (Ws) (D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor from the heated purge of the calibration standard.

D = $\frac{100 - \% \text{ moisture}}{100}$

Ws = Weight of sample added to the purge tube, in grams (g).

11.5.3 Medium Level Soil/Sediment Samples

The following equation is used for quantitation of medium level soil/sediment samples:

$$\text{Concentration ug/Kg (Dry weight basis)} = \frac{(Ax) (Is) (Vt) (1000) (Df)}{(Ais) (RRF) (Va) (Ws) (D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor from the ambient temperature purge of the calibration standard.

Vt = Total volume of the methanol extract in milliliters (mL).

NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial.

Va = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 uL) in micro liters (uL) added to reagent water for purging.

Ws = Weight of soil/sediment extracted, in grams (g).

D = $\frac{100 - \% \text{ moisture}}{100}$

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\text{ul most conc. extract used to make dilution} + \text{ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

(The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (Vt). The factor of 1,000 in the numerator converts the value of Vt from mL to uL.)

- 11.6** When quantitating the sample concentration of Xylenes (total), the areas of both the m & p Xylene peak and the o-Xylene peak are summed and the RRF determined using equation 11.2.1 are used. The concentration of each peak may be determined separately and then summed to determine the concentration of Xylene (total).
- 11.7** When quantitating the concentration of 1,2-Dichloroethene (total), the concentrations of the two isomers (cis and trans) are summed.
- 11.8** Secondary ion quantitation may be used if interferences (such as matrix effects) may cause a bias in quantitation.
- 11.9** If manual integration of any compound (including internal standards, system monitoring compounds, target or tentatively identified compounds) is required, the EICP of that compound will be provided. All manual integrations will be identified with an "m" and initialed and dated by the GC/MS analyst.
- 11.10 Tentatively Identified Compounds**
- 11.10.1** An estimated concentration for tentatively identified compounds will be determined using the equations described above for a given matrix using the total area counts of both the tentatively identified compound and the nearest internal standard which is free of interferences.

11.10.2 The RRF used to determine all concentrations of tentatively identified compounds will be an assumed RRF of one (1). The Chrome data system is set to a Q value of 85% and any tentatively identified compound with a Q value less than 85% will be named unknown.

11.10.3 All tentatively identified compounds will be qualified as "J" (estimated) and "N" (presumptive evidence).

11.11 System Monitoring Compounds

11.11.1 The recovery of all system monitoring compounds in samples, blanks matrix spikes and matrix spike duplicates, is calculated using equation:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

11.11.2 The recovery limits for each system monitoring compound are laboratory established on an annual basis. The recoveries must be within the criteria limits. If they fall outside criteria limits, the results must be evaluated and the sample reanalyzed, if necessary.

11.11.3 The relative retention time (RRT) of each system monitoring compound must be within the acceptance windows of ± 0.06 RRT.

11.12 Internal Standards

11.12.1 The internal standards of all samples, blanks, matrix spikes and matrix spike duplicates must be monitored. The EICP area of each internal standard must be within the range of -50.0 percent to 200.0 percent of those in the continuing calibration.

11.12.2 The relative retention time (RRT) of each internal standard must be within 0.5 minutes (30 seconds) of those in the continuing calibration.

11.13 Verification of Calculated Result

11.13.1 The laboratory analyst/data entry analyst will print out and review sample worksheets and hand calculate the result for positive hits, internal standards and surrogates for comparison to the LIMS calculated result. Corrective action will result, if needed.

12.0 Method Performance

Each analyst prior to sample analysis will perform 4 replicate QC check standards as an Initial Demonstration of Capability. The average recovery and standard deviation are calculated in the LIMS system and kept with each analyst's training file.

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

- 12.2.1** A one-time initial demonstration of performance for each individual method for both soils and water matrices must be generated.
- 12.2.2** This requires quadruplicate analysis of a mid-level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.
- 12.2.3** Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).
- 12.2.4** Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

- 12.3.1** The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 12.3.2** The following analyst validation information is maintained for this method in the laboratory QA files.
- 12.3.3** The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- 12.3.4** The analyst must read and understand this SOP.
- 12.3.5** The analyst must read and understand the Method used as reference for this SOP.
- 12.3.6** The analyst must complete a DOC or successfully analyze PT samples annually.
- 12.3.7** The analyst must complete the TestAmerica Quality Assurance Training.

13.0 **Pollution Control**

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

14.0 **Waste Management**

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

14.1 **Waste Streams Produced by the Method**

The following waste streams are produced when this method is carried out.

14.1.1 Spill Response: Any spills must be cleaned up immediately and handled correctly. Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

14.1.2 Aqueous waste generated from analysis: Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

14.1.3 Solvent waste generated from analysis: Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste. Waste receptacles are then taken to sample control where they are then properly disposed of.

14.1.4 Solid waste generated from analysis: Solid volatile analysis waste consists of soils and glass. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner. Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles

14.1.5 Expired Standards: Expired and used standards are stored in a laboratory approved metal waste receptacle labeled "BV". Waste receptacles are then taken to sample control where they are then properly disposed of.

15.0 **References / Cross-References**

- Method 8260C, "Test Methods for Evaluating Solid Waste"; SW846, Fourth Edition, August 2006.

16.0 **Method Modifications:** NA

17.0 Attachments

Table 1. Compounds Determined by Method 8260C
Table 2. BFB Key Ions and Ion Abundance Criteria
Table 3. Volume of Medium Level Extracts for Dilution
Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds
Table 5. Poor Performing Compounds
Table 5a. Defined CCV Poor performing compounds
Table 6. Minimum Response Factors
Table 7. Job Summary Check List (Page 1 & 2)
Tables 8-16. Composition of Stock Standards
Table 17. TestAmerica Buffalo GCMS VOA Dilution Calculation

18.0 Revision History

Revision 01, dated -13 January, 2015

- Section 1.1.1: Removed reference to 25mL purge analysis – no longer perform.
- Section 2.2: Removed reference to 25mL purge analysis.
- Section 3.6 Change batch to 18 field samples.
- Section 9.4.1.4 Added the read back of the low level standard for linear fit analytes.
- Section 7.3.1.2: Changed the standards to incorporate the new corporate formulations.
- Section 7.3.1.3: Changed the standards to incorporate the new add corporate formulations.
- Section 7.3.2.1&2: Changed the second source standards to incorporate the new corporate formulations.
- Section 7.3.3: Removed 1,4-difluorobenzene and added Fluorobenzene – new corporate mixes.
- Section 7.3.4: Added Dibromofluoromethane – new corporate mixes.
- Section 7.3.5: Removed Stock Matrix Spike Solution – do not use.
- Section 7.4(old): Removed whole section as it is not needed (secondary IS and Surrogate dilution standards).
- Section 7.4.1.2: Removed the 54 component reference and changed to the 8260 Mega mix reference.
- Section 7.4.1.2: Change the number of stocks from four to five.
- Section 7.4.2: Removed the 17 compound spike references and removed the verification in standard.
- Section 7.4.2.2: Changed the 200uL to 1000uL of the 8260 Mega mixes; four mixes to five.
- Section 7.4.5: Removed the 25mL purge curve reference. Reduced curves to 6 point calibrations for both waters and soils.
- Section 7.4.5.1.1: Changed 5mL water initial curve to 6 points. Removed the surrogate multipoint references.
- Section 7.4.5.1.2: Removed the multipoint surrogate calibration.
- Section 7.4.5.2.1: Changed 5mL soil initial curve to 6 points. Removed the surrogate multipoint references.
- Section 7.4.5.2.2: Removed the multipoint surrogate calibration.
- Section 7.4.6: Removed the 25mL continuing calibration standards.
- Section 8.5.1: Removed the 8uL of 50 ng/uL or 4uL of 100 ng/uL matrix spike standard for 25mL and 22uL of 50ng/uL. Also removed the concentrations related to 25mL purge.
- Section 8.5.2: Removed 5uL of 50ng/uL standard.
- Section 9.2.1: Removed 10ug/L for 25mL analysis.

- Section 9.2.2: Removed 10ug/L for 25mL analysis.
- Section 9.2.5: Removed the 17 compound spike references and removed the when required from the full spike.
- Section 9.4.1.2: Added the alternate quant ion statement.
- Section 9.4.1.4.2: Added ICV allowed at 50% for poor performing compounds.
- Section 9.4.2.2: Added CCV failure corrective actions.
- Section 9.5.5.1: Removed the 17 compound spike reference as we use full spike.
- Section 10.1.3: Removed the chart for 25mL Purge analysis and * for optional 6th point.
- Section 10.2.1: Removed 25mL purge amount.
- Section 10.2.4.2: Removed 25mL purge reference.
- Section 10.4.1: Removed 25mL purge reference.
- Section 10.6.3: Added the correct procedure for medium level soil prep for method 5035.
- Section 11.10.2: Added the Chrome setting for the 85% Q value and naming convention.
- Table 1: Added compounds to additional analytes amenable to 8260 analysis.
- Table 3: Change volume of extract header.
- Table 4: Removed 1,4-difluorobenzene and added Fluorobenzene and the correct primary and secondary ions.
- Table 5: Removed add compounds as we do not spike with adds and added Bromoform, Isobutyl alcohol and 2-methyl-2-propanol.
- Table 5a: Defined CCV poor performers.
- Table 8: Replaced gas mix certification.
- Table 9: Changed the 54 compound mix to 8260 Mega Mix and put in new certification.
- Table 10: Changed the title of table and the certifications.
- Table 11: Changed the title of table and the certifications.
- Table 12: Changed the title of table and the certifications.
- Table 13: Changed the certifications.
- Table 15: Changed the certifications.
- Table 16: Changed the certifications.

Initial Version

Table 1: Compounds Determined by Method 8260C

Compound	CAS No. ^b	Appropriate Technique					Direct Injection
		5030/5035	5031	5032	5021	5041	
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-03-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c

Compound	CAS No. ^b	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c

Compound	CAS No. ^b	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
1,4-Difluorobenzene (I.S.)	540-36-3	nd	nd	nd	nd	c	c
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	l	nd	nd	nd	nd	c

Compound	CAS No. ^b	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	l	nd	nd	nd	c
B-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	c
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	l	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-33	c	nd	c	c	c	c
Toluene-d8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluene	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethane	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
Xylene (Total)	1330-20-7	c	nd	c	c	c	c

c= Adequate response by this technique
 b= Chemical Abstract Services Registry Number
 pp= Poor purging efficiency resulting in high EQLs
 l= Inappropriate technique for this analyte

nd= Not determined
 surr= Surrogate
 IS= Internal Standard
 ht= Method analyte only when purged at 80 C

pc= Poor chromatographic behavior

The following compounds are also amenable to analysis by Method 8260:

Bromobenzene	1,3-Dichloropropane
n-Butylbenzene	2,2-Dichloropropane
sec-Butylbenzene	1,1-Dichloropropene
tert-Butylbenzene	p-Isopropyltoluene
Chloroacetonitrile	Methyl acrylate
1-Chlorobutane	Methyl-t-butyl ether
1-Chlorohexane	Pentafluorobenzene
2-Chlorotoluene	n-Propylbenzene
4-Chlorotoluene	1,2,3-Trichlorobenzene
Dibromofluoromethane	1,2,4-Trimethylbenzene
cis-1,2-Dichloroethene	1,3,5-Trimethylbenzene
Di-isopropyl ether	tert-butyl ethyl ether
tert-amyl methyl ether	benzyl chloride
isooctane	n-butyl acetate
2-methylnaphthalene	ethanol
t-amyl alcohol	

Table 2. BFB Key Ions and Ion Abundance Criteria

<u>m/z</u>	<u>Required Intensity (relative abundance)</u>
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

*Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

Table 3. Volume of Medium Level Extracts for Dilution (for a 5mL purge volume)

Dilution Factor	Volume of ML soil Extract
1	100ul
2	50ul
5	20ul
10	10ul
20	5ul
25	4ul
40	2.5ul
50	2ul
100	1ul
200	50ul of a 1/10 Dilution

Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40,39
Acrolein	56	55,58
Acrylonitrile	53	52,51
Allyl alcohol	57	58,39
Allyl chloride	76	41,39,78
Benzene	78	-
Benzyl chloride	91	126,65,128
Bromoacetone	136	43,138,93,95
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44,84,86,111
Chloroacetonitrile	48	75
Chlorobenzene	112	77,114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208,206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44,43,51,80
bis-(2-Chloroethyl) sulfide	109	111,158,160
2-Chloroethyl vinyl ether	63	65,106

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88,90,51
3-Chloropropionitrile	54	49,89,91
3-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene-d ₄	152	115,150
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
cis-1,4-Dichloro-2-butene	75	53,77,124,89
trans-1,4-Dichloro-2-butene	53	88,75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43,81,49
1,1-Dichloropropene	75	110,77
cis-1,3-Dichloropropene	75	77,39
trans-1,3-Dichloropropene	75	77,39
1,2,3,4-Diepoxybutane	55	57,56
Diethyl ether	74	45,59
1,4-Dioxane	88	58,43,57
Epichlorohydrin	57	49,62,51
Ethanol	31	45,27,46
Ethyl acetate	88	43,45,61
Ethylbenzene	91	106
Ethylene oxide	44	43,42
Ethyl methacrylate	69	41,99,86,114
Hexachlorobutadiene	225	223,227
Hexachloroethane	201	166,199,203
2-Hexanone	43	58,57,100
2-Hydroxypropionitrile	44	43,42,53
Iodomethane	142	127,141
Isobutyl alcohol	43	41,42,74
Isopropylbenzene	105	120
p-Isopropyl toluene	119	134,91
Malonitrile	66	39,65,38
Methacrylonitrile	41	67,39,52,66
Methyl acrylate	55	85

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl-t-butyl ether	73	57
Methylene chloride	84	86,49
Methyl ethyl ketone	72	43
Methyl iodide	142	127,141
Methyl methacrylate	69	41,100,39
4-Methyl-2-pentanone	100	43,58,85
Naphthalene	128	-
Nitrobenzene	123	51,77
2-Nitropropane	46	-
2-Picoline	93	66,92,78
Pentachloroethane	167	130,132,165,169
Propargyl alcohol	55	39,38,53
B-Propiolactone	42	43,44
Propionitrile (ethyl cyanide)	54	52,55,40
n-Propylamine	59	41,39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	97,130,132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromochloromethane-d2	51	131
Fluorobenzene	96	77
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115,150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174,176
Chloroform-d1	84	
Dibromofluoromethane	113	

Table 5. Poor Performing Compounds

Bromoform	Bromomethane
1,2-Dibromo-3-chloropropane (DBCP)	Carbon Disulfide
1,4-Dioxane	Chloroethane
2-Butanone (MEK)	Isobutyl Alcohol
2-Chloroethylvinyl ether	Dichlorodifluoromethane
2-Methyl-2-propanol	Iodomethane
4-Methyl-2-pentanone (MIBK)	Methyl Acetate
Acetone	trans-1,4-Dichloro-2-butene
Acrolein	

Table 5a. Defined CCV Poor Performing Compounds

Bromoform	Bromomethane
1,2-Dibromo-3-chloropropane (DBCP)	Chloroethane
1,4-Dioxane	Isobutyl Alcohol
2-Methyl-2-propanol	Dichlorodifluoromethane
Acetone	Methyl Acetate
Acrolein	trans-1,4-Dichloro-2-butene

Table 6. Minimum Relative Response Factors

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND
CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Table 6. Minimum Relative Response Factors continued...

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

^a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

^b Data provided by EPA Region III laboratory.

Table 7. Job Summary Check List



GCMS VOA Work Order Summary

GCMS VOA Data Review Checklist
Rev. 1 October 24, 2012

Work Order: _____ Method: _____

Work Order Due: _____

Sequence #1	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____
Sequence #2	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____
Sequence #3	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____
Sequence #4	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____
Sequence #5	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____
Sequence #6	Curve Batch# _____	Prep Batch# _____
Chrom WL _____	TALS Batch _____	Instrument _____ Date Created _____

Analyte Comments: _____

Sample Comments: _____

First Level Review: Initials: _____ Date: _____

Second Level Review: Initials: _____ Date: _____

Check Second Level Review:

- ___ Quantitative Accuracy
- ___ Calibration
- ___ QC Samples
- ___ Method and/or Quapp Specific QC Criteria
- ___ Manual Integrations Reviewed

Table 8. Gas Mixture (8260 List 1/std 3 gases)

RESTEK

110 Benner Circle
Belleville, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com



Certificate of Analysis

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 567645 Lot No.: A093341
Description: 8260 List 1 / Std #3 Gases
8260 List 1 / Std #3 Gases 2,000 ug/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2015 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)		
1	Dichlorodifluoromethane (CFC-12)	2,000.0 µg/mL	+/-	13.8716	µg/mL Gravimetric
	CAS # 75-71-8		+/-	25.2661	µg/mL Unstressed
	Purity 99%		+/-	28.2336	µg/mL Stressed
2	Chloromethane (methyl chloride)	1,999.8 µg/mL	+/-	13.9993	µg/mL Gravimetric
	CAS # 74-87-3		+/-	25.3348	µg/mL Unstressed
	Purity 99%		+/-	28.2945	µg/mL Stressed
3	Vinyl chloride	2,000.1 µg/mL	+/-	13.9615	µg/mL Gravimetric
	CAS # 75-01-4		+/-	25.3168	µg/mL Unstressed
	Purity 99%		+/-	28.2792	µg/mL Stressed
4	1,3-Butadiene	2,000.0 µg/mL	+/-	13.3773	µg/mL Gravimetric
	CAS # 106-99-0		+/-	24.9981	µg/mL Unstressed
	Purity 99%		+/-	27.9940	µg/mL Stressed
5	Bromomethane (methyl bromide)	2,000.1 µg/mL	+/-	14.2856	µg/mL Gravimetric
	CAS # 74-83-9		+/-	25.4963	µg/mL Unstressed
	Purity 99%		+/-	28.4359	µg/mL Stressed
6	Chloroethane (ethyl chloride)	2,000.0 µg/mL	+/-	13.2200	µg/mL Gravimetric
	CAS # 75-00-3		+/-	24.9143	µg/mL Unstressed
	Purity 99%		+/-	27.9151	µg/mL Stressed
7	Dichlorofluoromethane (CFC-21)	2,000.0 µg/mL	+/-	13.5174	µg/mL Gravimetric
	CAS # 75-43-4		+/-	25.0735	µg/mL Unstressed
	Purity 99%		+/-	28.0614	µg/mL Stressed
8	Trichlorofluoromethane (CFC-11)	1,999.9 µg/mL	+/-	13.1170	µg/mL Gravimetric
	CAS # 75-69-4		+/-	24.8590	µg/mL Unstressed
	Purity 99%		+/-	27.8696	µg/mL Stressed
Solvent:	P&T Methanol				
	CAS # 67-56-1				
	Purity 99%				

Table 9. 8260 Mega Mixture (8260 List 1/Std 1 mega mix)

RESTEK

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com



Certificate of Analysis

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 567641 Lot No.: A093581
Description: 8260 List 1 / Std #1 MegaMix
8260 List 1 / Std #1 MegaMix 1000-50,000 µg/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Diethyl ether (ethyl ether) CAS # 60-29-7 Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed
2	1,1,2-Trichlorotrifluoroethane (CFC-113) CAS # 76-13-1 Purity 97%	1,999.9 µg/mL	+/- 11.6279 µg/mL Gravimetric +/- 44.2519 µg/mL Unstressed +/- 44.4323 µg/mL Stressed
3	1,1-dichloroethane CAS # 75-35-4 Purity 98%	2,000.0 µg/mL	+/- 11.6281 µg/mL Gravimetric +/- 44.2527 µg/mL Unstressed +/- 44.4331 µg/mL Stressed
4	tert-Butanol (TBA) CAS # 75-65-0 Purity 99%	20,000.0 µg/mL	+/- 116.2756 µg/mL Gravimetric +/- 442.5291 µg/mL Unstressed +/- 444.3332 µg/mL Stressed
5	Iodomethane (methyl iodide) CAS # 74-88-4 Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed
6	Allyl chloride (3-chloropropene) CAS # 107-05-1 Purity 98%	2,000.0 µg/mL	+/- 11.6281 µg/mL Gravimetric +/- 44.2527 µg/mL Unstressed +/- 44.4331 µg/mL Stressed
7	Methyl acetate CAS # 79-20-9 Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 221.2646 µg/mL Unstressed +/- 222.1666 µg/mL Stressed
8	Carbon disulfide CAS # 75-15-0 Purity 98%	2,000.0 µg/mL	+/- 11.6281 µg/mL Gravimetric +/- 44.2527 µg/mL Unstressed +/- 44.4331 µg/mL Stressed
9	Methylene chloride (dichloromethane) CAS # 75-09-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed

10	Acrylonitrile CAS # 107-13-1 Purity 99%	20,000.0 µg/mL	+/- 116.2756 +/- 442.5291 +/- 444.3332	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
11	Methyl-tert-butyl ether (MTBE) CAS # 1634-04-4 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
12	cis-1,2-Dichloroethene CAS # 156-59-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
13	n-Hexane (C6) CAS # 110-54-3 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
14	1,1-Dichloroethane CAS # 75-34-3 Purity 98%	2,000.0 µg/mL	+/- 11.6281 +/- 44.2527 +/- 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
15	2,2-Dichloropropane CAS # 594-20-7 Purity 98%	2,000.0 µg/mL	+/- 11.6281 +/- 44.2527 +/- 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
16	trans-1,2-Dichloroethene CAS # 156-60-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
17	chloroform CAS # 67-66-3 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
18	Isobutanol (2-Methyl-1-propanol) CAS # 78-83-1 Purity 99%	50,000.0 µg/mL	+/- 290.6891 +/- 1,106.3228 +/- 1,110.8331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
19	Bromochloromethane CAS # 74-97-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
20	Tetrahydrofuran CAS # 109-99-9 Purity 99%	4,000.0 µg/mL	+/- 23.2563 +/- 88.5061 +/- 88.8670	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
21	1,1,1-trichloroethane CAS # 71-55-6 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
22	Cyclohexane CAS # 110-82-7 Purity 98%	2,000.0 µg/mL	+/- 11.6281 +/- 44.2527 +/- 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
23	1,1-Dichloropropene CAS # 563-58-6 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
24	carbon tetrachloride CAS # 56-23-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
25	n-Heptane (C7) CAS # 142-82-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
26	Benzene CAS # 71-43-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
27	1,2-Dichloroethane CAS # 107-06-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
28	Trichloroethene CAS # 79-01-6 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed

29	Methylcyclohexane CAS # 108-87-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
30	1,2-Dichloropropane CAS # 78-87-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
31	1,4-Dioxane CAS # 123-91-1 Purity 99%	40,000.0 µg/mL	+/- 232.5513 +/- 885.0582 +/- 888.6665	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
32	Dibromomethane CAS # 74-95-3 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
33	bromodichloromethane CAS # 75-27-4 Purity 97%	2,000.0 µg/mL	+/- 11.6284 +/- 44.2540 +/- 44.4344	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
34	cis-1,3-Dichloropropene CAS # 10061-01-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
35	Toluene CAS # 108-88-3 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
36	Ethyl methacrylate CAS # 97-63-2 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
37	trans-1,3-Dichloropropene CAS # 10061-02-6 Purity 97%	2,000.0 µg/mL	+/- 11.6284 +/- 44.2540 +/- 44.4344	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
38	1,1,2-Trichloroethane CAS # 79-00-5 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
39	1,3-Dichloropropane CAS # 142-28-9 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
40	Tetrachloroethene CAS # 127-18-4 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
41	dibromochloromethane CAS # 124-48-1 Purity 98%	2,000.0 µg/mL	+/- 11.6281 +/- 44.2527 +/- 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
42	1,2-Dibromoethane (EDB) CAS # 106-93-4 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
43	Chlorobenzene CAS # 108-90-7 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
44	1,1,1,2-Tetrachloroethane CAS # 630-20-6 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
45	m-Xylene CAS # 108-38-3 Purity 99%	1,000.0 µg/mL	+/- 5.8141 +/- 22.1265 +/- 22.2167	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
46	p-Xylene CAS # 106-42-3 Purity 99%	1,000.0 µg/mL	+/- 5.8141 +/- 22.1265 +/- 22.2167	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
47	o-Xylene CAS # 95-47-6 Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed

48	Ethylbenzene CAS # 100-41-4 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
49	Styrene CAS # 100-42-5 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
50	Isopropylbenzene (cumene) CAS # 98-82-8 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
51	bromoform CAS # 75-25-2 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
52	1,1,2,2-Tetrachloroethane CAS # 79-34-5 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
53	1,2,3-Trichloropropane CAS # 96-18-4 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
54	trans-1,4-dichloro-2-butene CAS # 110-57-6 Purity 98%	2,000.0 µg/mL	+/- 11,6281 +/- 44,2527 +/- 44,4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
55	n-Propylbenzene CAS # 103-65-1 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
56	Bromobenzene CAS # 108-86-1 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
57	1,3,5-Trimethylbenzene CAS # 108-67-8 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
58	2-Chlorotoluene CAS # 95-49-8 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
59	4-Chlorotoluene CAS # 106-43-4 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
60	tert-Butylbenzene CAS # 98-06-6 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
61	1,2,4-Trimethylbenzene CAS # 95-63-6 Purity 98%	2,000.0 µg/mL	+/- 11,6281 +/- 44,2527 +/- 44,4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
62	sec-Butylbenzene CAS # 135-98-8 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
63	4-Isopropyltoluene (p-Cymene) CAS # 99-87-6 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
64	1,3-Dichlorobenzene CAS # 541-73-1 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
65	1,4-Dichlorobenzene CAS # 106-46-7 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
66	n-Butylbenzene CAS # 104-51-8 Purity 99%	2,000.0 µg/mL	+/- 11,6282 +/- 44,2531 +/- 44,4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed

Table 10. 8260 Mega Mix (List 1/Standard 2,4 5 6)



110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

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This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 567642 Lot No.: A093365
Description: 8260 List 1 / Std #2 Ketones
8260 List 1 / Std #2 Ketones 10,000 ug/ml, P&T Methanol/Water (90:10), 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Acetone CAS # 67-64-1 Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 798.6896 µg/mL Unstressed +/- 799.0807 µg/mL Stressed
2	2-Butanone (MEK) CAS # 78-93-3 Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 798.6896 µg/mL Unstressed +/- 799.0807 µg/mL Stressed
3	4-Methyl-2-pentanone (MIBK) CAS # 108-10-1 Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 798.6896 µg/mL Unstressed +/- 799.0807 µg/mL Stressed
4	2-Hexanone CAS # 591-78-6 Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 798.6896 µg/mL Unstressed +/- 799.0807 µg/mL Stressed
Solvent:	P&T Methanol/Water (90:10) CAS # 67-56-1/7732-18-5 Purity 99%		



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Catalog No.: 567643 Lot No.: A093368
Description: 8260 List 1 / Std #4 2-ChloroethylVinyl Ether
8260 List 1 / Std #4 2-ChloroethylVinyl Ether 2,000 ug/ml, P&T Methanol,
1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)	
1	2-Chloroethyl vinyl ether	2,000.0 µg/mL	+/- 11.6282 µg/mL	Gravimetric
	CAS # 110-75-8		+/- 44.2531 µg/mL	Unstressed
	Purity 99%		+/- 44.4335 µg/mL	Stressed
Solvent:	P&T Methanol			
	CAS # 67-56-1			
	Purity 99%			

Tech Tips:

Degradation of tetrachloroethylene to pentachloroethane may occur if solutions containing 2-chloroethyl vinyl ether are combined with solutions that contain tetrachloroethylene.

Degradation of tetrachloroethylene to pentachloroethane may occur if solutions containing 2-chloroethyl vinyl ether are combined with solutions that contain tetrachloroethylene.

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This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 568720 Lot No.: A0100019
Description: 8260 List 1/Std #5 Acrolein High
8260 List 1/Std #5 Acrolein High 20,000 µg/mL, Water, 1 mL/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: April 30, 2014 Storage: 10°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Acrolein CAS # 107-02-8 Purity 99% (Lot 130830JLM)	20,000.7 µg/mL	±117,1083 µg/mL Gravimetric ±641,2843 µg/mL Unstressed ±745,4228 µg/mL Stressed

Solvent: Water
CAS # 7732-18-5
Purity 99%



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This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 567646 Lot No.: A099480
Description: 8260 List 1 / Std #5 Vinyl Acetate
8260 List 1 / Std #6 Vinyl Acetate 4000 ug/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: May 31, 2014 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Vinyl acetate CAS # 108-05-4 Purity 99% (Lot 131011JLM)	3,994.0 µg/mL	+/- 23.4390 µg/mL Gravimetric +/- 318.1798 µg/mL Unstressed +/- 318.3364 µg/mL Stressed

Solvent: P&T Methanol
CAS # 67-56-1
Purity 99%

Tech Tips:

Vinyl acetate is a volatile organic ester included in the target lists of several US EPA and other methods. Under acidic conditions, esters react with alcohols to form new esters (transesterification). Methanol-based mixes containing halogenated compounds are slightly acidic, so it is important to minimize exposure of vinyl acetate to mixes of halogenated compounds in methanol. For this reason, we offer vinyl acetate in individual solution, and suggest that it be introduced into the working level calibration solution immediately before use. This will minimize problems and ensure more consistent results.

Table 11. Second Source 8260 Mega Mix

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This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 567641.sec Lot No.: A093733
Description: 8260 List 1 / Std #1 MegaMix
8260 List 1 / Std #1 MegaMix 1,000-50,000 µg/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L., K=2)
1	Diethyl ether (ethyl ether) CAS # 60-29-7.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed
2	1,1,2-Trichlorotrifluoroethane (CFC-113) CAS # 76-13-1.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed
3	1,1-Dichloroethene CAS # 75-35-1.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed
4	tert-Butanol (TBA) CAS # 75-65-0.SEC Purity 99%	20,000.0 µg/mL	+/- 116.2756 µg/mL Gravimetric +/- 442.5291 µg/mL Unstressed +/- 444.3332 µg/mL Stressed
5	Iodomethane (methyl iodide) CAS # 74-88-4.SEC Purity 97%	2,000.0 µg/mL	+/- 11.6284 µg/mL Gravimetric +/- 44.2540 µg/mL Unstressed +/- 44.4344 µg/mL Stressed
6	Allyl chloride (3-chloropropene) CAS # 107-05-1.SEC Purity 98%	2,000.0 µg/mL	+/- 11.6281 µg/mL Gravimetric +/- 44.2527 µg/mL Unstressed +/- 44.4331 µg/mL Stressed
7	Methyl acetate CAS # 79-20-9.SEC Purity 99%	10,000.0 µg/mL	+/- 58.1378 µg/mL Gravimetric +/- 221.2646 µg/mL Unstressed +/- 222.1666 µg/mL Stressed
8	Carbon disulfide CAS # 75-15-0.SEC Purity 98%	2,000.0 µg/mL	+/- 11.6281 µg/mL Gravimetric +/- 44.2527 µg/mL Unstressed +/- 44.4331 µg/mL Stressed
9	Methylene chloride (dichloromethane) CAS # 75-09-2.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 µg/mL Gravimetric +/- 44.2531 µg/mL Unstressed +/- 44.4335 µg/mL Stressed

10	Acrylonitrile CAS # 107-13-1.SEC Purity 99%	20,000.0	µg/mL	+/-	116.2756	µg/mL	Gravimetric
				+/-	442.5291	µg/mL	Unstressed
				+/-	444.3332	µg/mL	Stressed
11	Methyl-tert-butyl ether (MTBE) CAS # 1634-04-4.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
12	cis-1,2-Dichloroethene CAS # 156-59-2.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
13	n-Hexane (C6) CAS # 110-54-3.SEC Purity 98%	2,000.1	µg/mL	+/-	11.6286	µg/mL	Gravimetric
				+/-	44.2549	µg/mL	Unstressed
				+/-	44.4353	µg/mL	Stressed
14	1,1-Dichloroethane CAS # 75-34-3.SEC Purity 97%	2,000.0	µg/mL	+/-	11.6284	µg/mL	Gravimetric
				+/-	44.2540	µg/mL	Unstressed
				+/-	44.4344	µg/mL	Stressed
15	2,2-Dichloropropane CAS # 594-20-7.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
16	trans-1,2-Dichloroethene CAS # 156-60-5.SEC Purity 97%	2,000.0	µg/mL	+/-	11.6284	µg/mL	Gravimetric
				+/-	44.2540	µg/mL	Unstressed
				+/-	44.4344	µg/mL	Stressed
17	Chloroform CAS # 67-66-3.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
18	Isobutanol (2-Methyl-1-propanol) CAS # 78-83-1.SEC Purity 99%	50,000.0	µg/mL	+/-	290.6891	µg/mL	Gravimetric
				+/-	1,106.3228	µg/mL	Unstressed
				+/-	1,110.8331	µg/mL	Stressed
19	Bromochloromethane CAS # 74-97-5.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
20	Tetrahydrofuran CAS # 109-99-9.SEC Purity 99%	4,000.0	µg/mL	+/-	23.2563	µg/mL	Gravimetric
				+/-	88.5061	µg/mL	Unstressed
				+/-	88.8670	µg/mL	Stressed
21	1,1,1-Trichloroethane CAS # 71-55-6.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
22	Cyclohexane CAS # 110-82-7.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
23	1,1-Dichloropropene CAS # 563-58-6.SEC Purity 98%	2,010.5	µg/mL	+/-	11.6890	µg/mL	Gravimetric
				+/-	44.4847	µg/mL	Unstressed
				+/-	44.6661	µg/mL	Stressed
24	Carbon tetrachloride CAS # 56-23-5.SEC Purity 98%	2,000.1	µg/mL	+/-	11.6286	µg/mL	Gravimetric
				+/-	44.2549	µg/mL	Unstressed
				+/-	44.4353	µg/mL	Stressed
25	n-Heptane (C7) CAS # 142-82-5.SEC Purity 99%	2,000.1	µg/mL	+/-	11.6288	µg/mL	Gravimetric
				+/-	44.2553	µg/mL	Unstressed
				+/-	44.4357	µg/mL	Stressed
26	Benzene CAS # 71-43-2.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
27	1,2-Dichloroethane CAS # 107-06-2.SEC Purity 99%	2,000.0	µg/mL	+/-	11.6282	µg/mL	Gravimetric
				+/-	44.2531	µg/mL	Unstressed
				+/-	44.4335	µg/mL	Stressed
28	Trichloroethene CAS # 79-01-6.SEC Purity 98%	2,000.1	µg/mL	+/-	11.6286	µg/mL	Gravimetric
				+/-	44.2549	µg/mL	Unstressed
				+/-	44.4353	µg/mL	Stressed

29	Methylcyclohexane CAS # 108-87-2.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
30	1,2-Dichloropropane CAS # 78-87-5.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
31	1,4-Dioxane CAS # 123-91-1.SEC Purity 99%	40,000.0 µg/mL	+/- 232.5513 +/- 885.0582 +/- 888.6665	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
32	Dibromomethane CAS # 74-95-3.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
33	Bromodichloromethane CAS # 75-27-4.SEC Purity 97%	2,000.1 µg/mL	+/- 11.6290 +/- 44.2562 +/- 44.4366	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
34	cis-1,3-Dichloropropene CAS # 10061-01-5.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
35	Toluene CAS # 108-88-3.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
36	Ethyl methacrylate CAS # 97-63-2.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
37	trans-1,3-Dichloropropene CAS # 10061-02-5.SEC Purity 98%	2,000.0 µg/mL	+/- 11.6281 +/- 44.2527 +/- 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
38	1,1,2-Trichloroethane CAS # 79-00-5.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
39	1,3-Dichloropropane CAS # 142-28-9.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
40	Tetrachloroethane CAS # 127-18-4.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
41	Dibromochloromethane CAS # 124-48-1.SEC Purity 97%	2,000.1 µg/mL	+/- 11.6290 +/- 44.2562 +/- 44.4366	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
42	1,2-Dibromoethane (EDB) CAS # 106-93-4.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
43	Chlorobenzene CAS # 108-90-7.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
44	1,1,1,2-Tetrachloroethane CAS # 630-20-6.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
45	m-Xylene CAS # 108-38-3.SEC Purity 99%	1,000.0 µg/mL	+/- 5.8141 +/- 22.1265 +/- 22.2167	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
46	p-Xylene CAS # 106-42-3.SEC Purity 99%	1,000.0 µg/mL	+/- 5.8141 +/- 22.1265 +/- 22.2167	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
47	o-Xylene CAS # 95-47-6.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed

48	Ethylbenzene CAS # 100-41-4.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
49	Styrene CAS # 100-42-5.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
50	Isopropylbenzene (cumene) CAS # 98-82-8.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
51	Bromoform CAS # 75-25-2.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
52	1,1,2,2-Tetrachloroethane CAS # 79-34-5.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
53	1,2,3-Trichloropropane CAS # 96-18-4.SEC Purity 98%	2,000.0 µg/mL	+/-	11.6281 44.2527 44.4331	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
54	trans-1,4-Dichloro-2-butene CAS # 110-57-6.SEC Purity 97%	2,000.0 µg/mL	+/-	11.6284 44.2540 44.4344	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
55	n-Propylbenzene CAS # 103-65-1.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
56	Bromobenzene CAS # 108-86-1.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
57	1,3,5-Trimethylbenzene CAS # 108-67-8.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
58	2-Chlorotoluene CAS # 95-49-8.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
59	4-Chlorotoluene CAS # 106-43-4.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
60	tert-Butylbenzene CAS # 98-06-6.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
61	1,2,4-Trimethylbenzene CAS # 95-63-6.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
62	sec-Butylbenzene CAS # 135-98-8.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
63	4-Isopropyltoluene (p-cymene) CAS # 99-87-6.SEC Purity 96%	2,000.1 µg/mL	+/-	11.6285 44.2545 44.4349	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
64	1,3-Dichlorobenzene CAS # 541-73-1.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
65	1,4-Dichlorobenzene CAS # 106-46-7.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
66	n-Butylbenzene CAS # 104-51-8.SEC Purity 99%	2,000.0 µg/mL	+/-	11.6282 44.2531 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed

67	1,2-Dichlorobenzene CAS # 95-50-1.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
68	1,2-Dibromo-3-chloropropane CAS # 96-12-8.SEC Purity 97%	2,000.0 µg/mL	+/- 11.6284 +/- 44.2540 +/- 44.4344	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
69	1,2,4-Trichlorobenzene CAS # 120-82-1.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
70	Hexachlorobutadiene CAS # 87-68-3.SEC Purity 97%	2,000.0 µg/mL	+/- 11.6284 +/- 44.2540 +/- 44.4344	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
71	Naphthalene CAS # 91-20-3.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
72	1,2,3-Trichlorobenzene CAS # 87-61-6.SEC Purity 99%	2,000.0 µg/mL	+/- 11.6282 +/- 44.2531 +/- 44.4335	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
Solvent: F&T Methanol					
CAS # 67-56-1					
Purity 99%					

Column:60m x .25mm x 1.4µm
Rtx502.2 (cat.#16914)**Carrier Gas:**

helium-constant pressure 30 psi

Temp. Program:40°C (hold 5 min.) to 240°C
@ 5°C/min. (hold 10 min.)**Inj. Temp:**

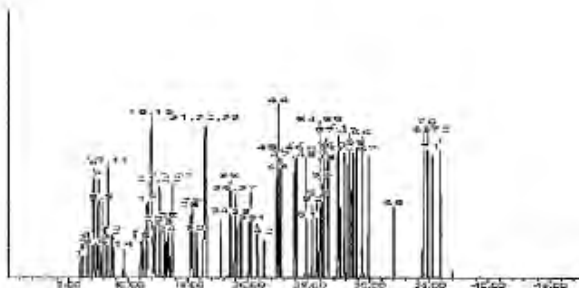
200°C

Det. Temp:

250°C

Det. Type:

MSD



Jennifer L. Polino
Jennifer L. Polino - QC Analyst

Date Passed: 01-Mar-2012 Balance: 1127510105

Manufactured under Rostek's ISO 9001:2008
Registered Quality System
Certificate #FIM 80397

Table12. Second Source 8260 Mega Mix

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Catalog No.: 567645.sec Lot No.: A093618
Description: 8280 List 1 / Std #3 Gases
8260 List 1 / Std #3 Gases 2,000 ug/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2015 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L., K=2)		
1	Dichlorodifluoromethane (CFC-12)	2,001.8 µg/mL	+/-	11.3948	µg/mL Gravimetric
	CAS # 75-71-8, SEC		+/-	24.0125	µg/mL Unstressed
	Purity 99%		+/-	27.1228	µg/mL Stressed
2	Chloromethane (methyl chloride)	2,000.6 µg/mL	+/-	11.1434	µg/mL Gravimetric
	CAS # 74-87-3, SEC		+/-	23.8835	µg/mL Unstressed
	Purity 99%		+/-	27.0052	µg/mL Stressed
3	Vinyl chloride	1,996.5 µg/mL	+/-	13.5847	µg/mL Gravimetric
	CAS # 75-01-4, SEC		+/-	25.0789	µg/mL Unstressed
	Purity 99%		+/-	28.0563	µg/mL Stressed
4	1,3-Butadiene	2,003.5 µg/mL	+/-	10.3715	µg/mL Gravimetric
	CAS # 106-99-0, SEC		+/-	23.5601	µg/mL Unstressed
	Purity 99%		+/-	26.7280	µg/mL Stressed
5	Bromomethane (methyl bromide)	2,001.5 µg/mL	+/-	12.1569	µg/mL Gravimetric
	CAS # 74-83-9, SEC		+/-	24.3811	µg/mL Unstressed
	Purity 99%		+/-	27.4489	µg/mL Stressed
6	Chloroethane (ethyl chloride)	1,997.2 µg/mL	+/-	13.6408	µg/mL Gravimetric
	CAS # 75-00-3, SEC		+/-	25.1155	µg/mL Unstressed
	Purity 99%		+/-	28.0910	µg/mL Stressed
7	Dichlorofluoromethane (CFC-21)	2,004.9 µg/mL	+/-	12.2390	µg/mL Gravimetric
	CAS # 75-43-4, SEC		+/-	24.4529	µg/mL Unstressed
	Purity 99%		+/-	27.5224	µg/mL Stressed
8	Trichlorofluoromethane (CFC-11)	2,001.9 µg/mL	+/-	16.1788	µg/mL Gravimetric
	CAS # 75-69-4, SEC		+/-	26.6190	µg/mL Unstressed
	Purity 99%		+/-	29.4557	µg/mL Stressed
Solvent: P&T Methanol					
CAS # 67-56-1					
Purity 99%					



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Catalog No.: 567643.sec Lot No.: A093471
Description: 8260 List 1 / Std #4 2-Chloroethyl Vinyl Ether
8260 List 1 / Std #4 2-Chloroethyl Vinyl Ether 2,000 ug/ml, P&T Methanol,
1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	2-Chloroethyl vinyl ether	2,000.0 µg/mL	+/- 11,6550 µg/mL Gravimetric
	CAS # 110-75-8, SEC		+/- 44,2601 µg/mL Unstressed
	Purity 99%		+/- 44,4405 µg/mL Stressed
Solvent:	P&T Methanol		
	CAS # 67-56-1		
	Purity 99%		

Tech Tips:

Degradation of tetrachloroethylene to pentachloroethane may occur if solutions containing 2-chloroethyl vinyl ether are combined with solutions that contain tetrachloroethylene.

Degradation of tetrachloroethylene to pentachloroethane may occur if solutions containing 2-chloroethyl vinyl ether are combined with solutions that contain tetrachloroethylene.



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Catalog No.: 567644.sec Lot No.: AQ98699
Description: 8260 List 1 / Std # 5 Acrolein
8260 List 1 / Std # 5 Acrolein 5,000 ug/ml, Water, 1 mL/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 28, 2014 Storage: 10°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)	
1	Acrolein	5,006.2 µg/mL	+/- 29.3790 µg/mL	Gravimetric
	CAS # 107-02-8 SEC (Lot 1506200)		+/- 131.3997 µg/mL	Unstressed
	Purity 97%		+/- 165.1986 µg/mL	Stressed

Solvent: Water
CAS # 7732-18-5
Purity 99%

SS Acrolein Mix - 00003 thru 00006



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Catalog No.: 567646.SEC Lot No.: A098982
Description: 8260 List 1 / Std #6 Vinyl Acetate
8260 List 1 / Std #6 Vinyl Acetate 4000 ug/ml, P&T Methanol, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 28, 2014 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L., K=2)
1	Vinyl acetate	4,003.0 µg/mL	+/- 23.4918 µg/mL (gravimetric)
	CAS # 108-05-4.SEC (Lot GPTIF)		+/- 318.8968 µg/mL Unstressed
	Purity 99%		+/- 319.0538 µg/mL Stressed

Solvent: P&T Methanol
CAS # 67-56-1
Purity 99%

Tech Tips:

Vinyl acetate is a volatile organic ester included in the target lists of several US EPA and other methods. Under acidic conditions, esters react with alcohols to form new esters (transesterification). Methanol-based mixes containing halogenated compounds are slightly acidic, so it is important to minimize exposure of vinyl acetate to mixes of halogenated compounds in methanol. For this reason, we offer vinyl acetate in individual solution, and suggest that it be introduced into the working level calibration solution immediately before use. This will minimize problems and ensure more consistent results.

Table 13. 8260 Add Mixture



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Catalog No. : 568722 Lot No.: A0100262

Description : 8260 List 2/ Std #1 Additions (2014)
8260 List 2/ Std #1 Additions (2014) 2,000-50,000 µg/ml, P&T Methanol,
1 ml/ampul

Container Size : 2 mL Pkg Amt: > 1 mL

Expiration Date : June 30, 2015 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	2-Propanol (isopropanol)	20,007.0 µg/mL	+/- 117.1454 µg/mL Gravimetric
	CAS # 67-63-0 (Lot SHBC5752V)		+/- 1,064.8186 µg/mL Unstressed
	Purity 99%		+/- 1,065.9927 µg/mL Stressed
2	Chloroprene (2-chloro-1,3-butadiene)	2,000.0 µg/mL	+/- 32.2441 µg/mL Gravimetric
	CAS # 126-99-8 (Lot 130611JLM)		+/- 110.6029 µg/mL Unstressed
	Purity 99%		+/- 110.7159 µg/mL Stressed
3	Ethyl acetate	4,002.0 µg/mL	+/- 23.4860 µg/mL Gravimetric
	CAS # 141-78-6 (Lot SHBD3394V)		+/- 213.0615 µg/mL Unstressed
	Purity 99%		+/- 213.2364 µg/mL Stressed
4	Methacrylonitrile	20,000.5 µg/mL	+/- 117.1073 µg/mL Gravimetric
	CAS # 126-98-7 (Lot 2194000)		+/- 1,064.4727 µg/mL Unstressed
	Purity 99%		+/- 1,065.5464 µg/mL Stressed
5	2,2,4-Trimethylpentane (isooctane)	2,004.5 µg/mL	+/- 11.7635 µg/mL Gravimetric
	CAS # 540-84-1 (Lot SHBB2470V)		+/- 106.6871 µg/mL Unstressed
	Purity 99%		+/- 106.8047 µg/mL Stressed
6	1-Butanol	50,001.0 µg/mL	+/- 292.7518 µg/mL Gravimetric
	CAS # 71-36-3 (Lot SHBC1840V)		+/- 2,661.1667 µg/mL Unstressed
	Purity 99%		+/- 2,664.1010 µg/mL Stressed
7	1,4-Difluorobenzene	2,006.5 µg/mL	+/- 11.7753 µg/mL Gravimetric
	CAS # 540-36-3 (Lot 13105AO)		+/- 106.7935 µg/mL Unstressed
	Purity 99%		+/- 106.9112 µg/mL Stressed
8	Ethyl acrylate	2,005.5 µg/mL	+/- 11.7694 µg/mL Gravimetric
	CAS # 140-88-5 (Lot 10129902)		+/- 106.7403 µg/mL Unstressed
	Purity 99%		+/- 106.8580 µg/mL Stressed

9	Methyl methacrylate CAS # 80-62-6 Purity 99%	(Lot MKBK0839V)	4,003.0	µg/mL	+/- +/- +/-	23.4918 213.0548 213.2897	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
10	2-Nitropropane CAS # 79-46-9 Purity 97%	(Lot BCBJ4343V)	4,006.6	µg/mL	+/- +/- +/-	23.5129 213.2456 213.4807	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
11	Butyl acetate CAS # 123-86-4 Purity 99%	(Lot SHBC9340V)	2,001.0	µg/mL	+/- +/- +/-	11.7430 106.5008 106.6182	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
12	1-Chlorohexane CAS # 544-10-5 Purity 99%	(Lot 05107LK)	2,007.5	µg/mL	+/- +/- +/-	11.7811 106.8467 106.9645	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
13	1,2,3-Trimethylbenzene CAS # 526-73-8 Purity 97%	(Lot 8776.05-10)	2,004.0	µg/mL	+/- +/- +/-	11.7607 106.6615 106.7791	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
14	Benzyl chloride CAS # 100-44-7 Purity 99%	(Lot 20396BK)	2,009.0	µg/mL	+/- +/- +/-	11.7899 106.9266 107.0445	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
15	1,3,5-Trichlorobenzene CAS # 108-70-3 Purity 99%	(Lot 11319AS)	2,000.0	µg/mL	+/- +/- +/-	11.7371 106.4475 106.5649	µg/mL µg/mL µg/mL	Gravimetric Unstressed Stressed
Solvent:								
	P&T Methanol CAS # 67-56-1 Purity 99%							



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Catalog No. : 567719 Lot No.: A093359
Description : 8260 List 2 / Std #2
8260 List 2 / Std #2 2,000 ug/ml, P&T Methanol, 1 ml/ampul
Container Size : 2 mL Pkg Amt: > 1 mL
Expiration Date : February 2015 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (85% C.L.; K=2)
1	Pentachlorocyclohexane	2,000.0 µg/mL	+/- 11.6550 µg/mL Gravimetric
	CAS # 76-01-7		+/- 24.1265 µg/mL Unstressed
	Purity 99%		+/- 27.2132 µg/mL Stressed
2	2-Methylnaphthalene	1,999.9 µg/mL	+/- 11.6546 µg/mL Gravimetric
	CAS # 91-57-6		+/- 24.1156 µg/mL Unstressed
	Purity 96%		+/- 27.2121 µg/mL Stressed
Solvent: P&T Methanol			
CAS # 67-56-1			
Purity 99%			



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Catalog No.: 567648 Lot No.: A093361
Description: 8260 List 2 / Std #3 Cyclohexanone
8260 List 2 / Std #3 Cyclohexanone 20,000 ug/ml, Water, 1 mL/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: February 2016 Storage: 10°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)		
1	Cyclohexanone	20,000.0 µg/mL	+/- 116.2756	µg/mL	Gravimetric
	CAS # 108-94-1		+/- 1,597.3791	µg/mL	Unstressed
	Purity 99%		+/- 1,598.1615	µg/mL	Stressed
Solvent:	Water				
	CAS # 7732-18-5				
	Purity 99%				

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Catalog No.: 568723 Lot No.: A099930
Description: 8260 List 3/ Std#1 Polar Additions
8260 List 3/ Std#1 Polar Additions 2,000-100,000 µg/ml, 1 ml/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: December 31, 2015 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound			Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)			
1	Ethanol			100,355.6 µg/mL	+/-	586.9883	µg/mL	Gravimetric
	CAS #	64-17-5	(Lot SHBC8676V)		+/-	3,493.5733	µg/mL	Unstressed
	Purity	99%			+/-	3,613.2792	µg/mL	Stressed
2	Acetonitrile			20,015.9 µg/mL	+/-	117.1976	µg/mL	Gravimetric
	CAS #	75-05-8	(Lot SHB3177V)		+/-	697.4888	µg/mL	Unstressed
	Purity	98%			+/-	721.3879	µg/mL	Stressed
3	Diisopropyl ether (DIPE)			2,001.6 µg/mL	+/-	11.7465	µg/mL	Gravimetric
	CAS #	108-20-3	(Lot SHB86268V)		+/-	69.7537	µg/mL	Unstressed
	Purity	99%			+/-	72.1435	µg/mL	Stressed
4	Ethyl-tert-butyl ether (ETBE)			2,008.4 µg/mL	+/-	11.7864	µg/mL	Gravimetric
	CAS #	637-92-3	(Lot MKBP5984V)		+/-	69.9907	µg/mL	Unstressed
	Purity	99%			+/-	72.3885	µg/mL	Stressed
5	Propionitrile			20,039.6 µg/mL	+/-	117.3363	µg/mL	Gravimetric
	CAS #	107-12-0	(Lot BCBK0700V)		+/-	698.3142	µg/mL	Unstressed
	Purity	99%			+/-	722.2416	µg/mL	Stressed
6	tert-Amyl alcohol			20,035.2 µg/mL	+/-	117.3105	µg/mL	Gravimetric
	CAS #	75-85-4	(Lot STBB1898V)		+/-	698.1609	µg/mL	Unstressed
	Purity	99%			+/-	722.0831	µg/mL	Stressed
7	tert-Amyl methyl ether (TAME)			2,005.6 µg/mL	+/-	11.7700	µg/mL	Gravimetric
	CAS #	994-05-8	(Lot QS10284V)		+/-	69.8931	µg/mL	Unstressed
	Purity	99%			+/-	72.2876	µg/mL	Stressed

RESTEK CERTIFIED REFERENCE MATERIAL

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (14)353-1309

www.restek.com

Certificate of Analysis



FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No. : 568111 Lot No.: A095258
Description : Buffalo Add 2 Mix A
Buffalo Add 2 Mix A 5,000 µg/ml, P&T Methanol, 1 ml/ampul
Container Size : 2 mL Pkg Amt: > 1 mL
Expiration Date : May 31, 2014 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Propylene oxide CAS # 75-56-9 Purity 98% (Lot 85396FIMV)	5,000.0 µg/mL	+/- 29.3426 µg/mL Gravimetric +/- 110.7037 µg/mL Unstressed +/- 111.1544 µg/mL Stressed

Solvent: P&T Methanol
CAS # 67-56-1
Purity 99%

RESTEK CERTIFIED REFERENCE MATERIAL

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)358-1888
Fax: (814)353-1309

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Certificate of Analysis



FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 568112 Lot No.: A095252
Description: Buffalo Add 2 Mix B
Buffalo Add 2 Mix B, 1,000 - 20,000 µg/mL, P&T Methanol, 1 mL/ampul
Container Size: 2 mL Pkg Amt: > 1 mL
Expiration Date: November 30, 2014 Storage: 0°C or colder

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)
1	Chlorodifluoromethane (CFC-22) CAS # 75-45-6 Purity 99% (Lot Q162-44)	1,997.9 µg/mL	+/- 32.4219 µg/mL, Gravimetric +/- 53.5775 µg/mL, Unstressed +/- 53.7263 µg/mL, Stressed
2	Acetaldehyde dimethyl acetal CAS # 534-15-6 Purity 99% (Lot 01604PC)	5,000.0 µg/mL	+/- 29.3428 µg/mL, Gravimetric +/- 110.7046 µg/mL, Unstressed +/- 111.1553 µg/mL, Stressed
3	Isri-Amyl alcohol CAS # 75-85-4 Purity 99% (Lot STBB1898V)	20,001.0 µg/mL	+/- 117.1103 µg/mL, Gravimetric +/- 442.7697 µg/mL, Unstressed +/- 444.5731 µg/mL, Stressed
4	3-Chlorobenzotrifluoride CAS # 98-15-7 Purity 99% (Lot 21324DO)	1,003.0 µg/mL	+/- 5.9575 µg/mL, Gravimetric +/- 22.2264 µg/mL, Unstressed +/- 22.3167 µg/mL, Stressed
5	4-Chlorobenzotrifluoride CAS # 98-56-6 Purity 99% (Lot 08507BO)	1,003.0 µg/mL	+/- 5.9575 µg/mL, Gravimetric +/- 22.2264 µg/mL, Unstressed +/- 22.3167 µg/mL, Stressed
6	2-Chlorobenzotrifluoride CAS # 88-16-4 Purity 99% (Lot B0316DO)	1,003.0 µg/mL	+/- 5.9575 µg/mL, Gravimetric +/- 22.2264 µg/mL, Unstressed +/- 22.3167 µg/mL, Stressed
7	3-Chlorotoluene CAS # 108-41-8 Purity 99% (Lot 13528LX)	1,003.0 µg/mL	+/- 5.9575 µg/mL, Gravimetric +/- 22.2264 µg/mL, Unstressed +/- 22.3167 µg/mL, Stressed
8	Dicyclopentadiene CAS # 77-73-6 Purity 98% (Lot 14218BB)	999.6 µg/mL	+/- 5.9373 µg/mL, Gravimetric +/- 22.1510 µg/mL, Unstressed +/- 22.2410 µg/mL, Stressed

Table 14. BFB Standard

MVSL 3 1-710



CERTIFICATE OF ANALYSIS
FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30067
Description: 4-Bromofluorobenzene Standard
Expiration Date: January 2010

Lot No.: A036650
Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	1-Bromo-4-fluorobenzene (BFB)	460-00-4	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x 53mm x 3.0um
Rtx-502.2 (cat.#10910)

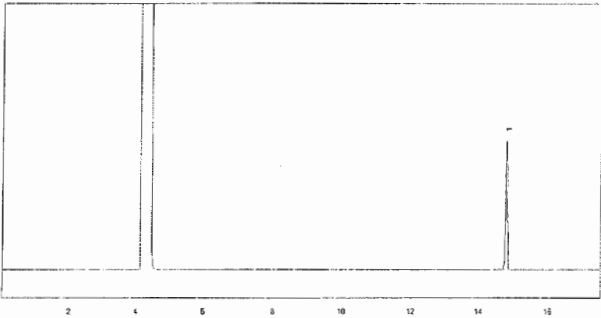
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
50°C to 240°C @ 10°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: MEW

John J. Judge
John J. Judge - O.R. Analyst



¹ Expiration date of the unopened ampul stored at recommended temperature.
² Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
³ Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (7 mass levels).
⁴ Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Table 15. Internal Standard Mixture



110 Banner Circle
Bellaire, PA 15823-8812
Tel: (300)356-1888
Fax: (814)353-1308
www.restek.com

CERTIFIED REFERENCE MATERIAL

Certificate of Analysis





FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.
This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 30241

Description: B26DA Internal Standard Mix
B26DA Internal Standard Mix 2,500 µg/mL, P&T Methanol, 1mL/ampul

Container Size: 2 mL

Expiration Date: November 30, 2018

Lot No.: A099180

Pkg Amt: > 1 mL

Storage: 0°C or colder

CERTIFIED VALUES

Index	Compound	Grav. Conc. (weight/volume)	Expected Uncertainty (95% CL, K=2)
1	Fluorobenzene CAS # 162-05-6 Purity 99% (Lot 1180033)	2,501.9 µg/mL	+/- 14.5405 µg/mL, Gravimetric, Unmeasured +/- 26.5347 µg/mL, Stressed +/- 30.8803 µg/mL, Stressed
2	Chlorobenzene-d5 CAS # 1114-55-4 Purity 99% (Lot PR-22736)	2,500.2 µg/mL	+/- 14.5364 µg/mL, Gravimetric, Unmeasured +/- 26.5365 µg/mL, Stressed +/- 30.8295 µg/mL, Stressed
3	1,4-Dichlorobenzene-d4 CAS # 3855-82-1 Purity 99% (Lot PR-18485)	2,501.5 µg/mL	+/- 14.5405 µg/mL, Gravimetric, Unmeasured +/- 26.5347 µg/mL, Stressed +/- 30.8803 µg/mL, Stressed

Solvent: P&T Methanol
CAS # 67-56-1
Purity 99%

Table 16. Surrogate Mixture



110 Banner Circle
Bellefonte, PA 16823-8912
Tel: (800)356-1888
Fax: (814)353-1308
www.restek.com





Certificate of Composition

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.
This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.:	567650	Lot No.:	A092213
Description:	8260R24 Surrogate Mix		
	8260R24 Surrogate Mix 2500 µg/mL, P&T Method, 5 mL/ampul		
Container Size:	5 mL	Pkg Amt:	> 5 mL
Expiration Date:	December 2017	Storage:	0°C or colder

CERTIFIED VALUES

Serial Order	Compound	Gross Conc. (µg/g or volume)	±	Relative Uncertainty (95% CL, K=2)	Assessment
1	Dibenzofluorenone CAS # 1861-53-7 Purity 99%	2,500.0 µg/mL	+/-	14.6711 µg/mL 30.2008 µg/mL 34.0606 µg/mL	Gravimetric Uncertained Stressed
2	1,2-Dichlorobenzene-d4 CAS # 17060-07-0 Purity 99%	2,500.0 µg/mL	+/-	14.6711 µg/mL 30.2008 µg/mL 34.0606 µg/mL	Gravimetric Uncertained Stressed
3	Toluene-d8 CAS # 20317-26-5 Purity 99%	2,500.0 µg/mL	+/-	14.6711 µg/mL 30.2008 µg/mL 34.0606 µg/mL	Gravimetric Uncertained Stressed
4	1-Bromo-4-fluorobenzene (BFB) CAS # 460-06-6 Purity 99%	2,500.0 µg/mL	+/-	14.6711 µg/mL 30.2008 µg/mL 34.0606 µg/mL	Gravimetric Uncertained Stressed
Solvent:	P&T Method CAS # 67-56-1 Purity 99%				

Page 1 of 3

TestAmerica Buffalo GCMS VOA Dilution Calculation

Table 17:

5mL and 25mL Water	2	(25mL/50mL)/5mL P&T
	4	(12.5mL/50mL)/5mL P&T
	5	(10mL/50mL)/5mL P&T
	8	(6.25mL/50mL)/5mL P&T
	10	(5mL/50mL)/5mL P&T
	20	(2.5mL/50mL)/5mL P&T
	25	(2mL/50mL)/5mL P&T
	40	(1.25mL/50mL)/5mL P&T
	50	(1mL/50mL)/5mL P&T
	80	(625uL/50mL)/5mL P&T
	100	(500uL/50mL)/5mL P&T
	125	(400uL/50mL)/5mL P&T
	200	(250uL/50mL)/5mL P&T
	400	(125uL/50mL)/5mL P&T
	500	(100uL/50mL)/5mL P&T
	800	(62.5uL/50mL)/5mL P&T
	1,000	(50uL/50mL)/5mL P&T
	2,000	(25uL/50mL)/5mL P&T
	4,000	(12.5uL/50mL)/5mL P&T
	5,000	(10uL/50mL)/5mL P&T
	8,000	(6.25uL/50mL)/5mL P&T

NOTE: 1. Primary dilutions are contained within the innermost parentheses. Any dilutions above 8000x are serial dilutions. The 50mL volumes are transferred into a 40mL Voa vial and contain zero headspace.

2. If the analyst does not see the dilution listed on the work instruction; the dilution performed must be indicated on the raw data, including the dilution factor, in the "sample Info": filed of the quantitation report.

**Toxicity Characteristic Leaching Procedure (TCLP)
EPA Method 1311**

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):



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Department Manager



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Distributed To: _____

1.0 Scope and Application

If a total analysis of any of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is applicable to liquid, solid and multiphasic wastes.

Reporting Limit: N/A

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

The wastes are initially characterized and defined by matrix (liquid, solid or mixed phase) and by pH. This preliminary characterization determines the type of TCLP extraction procedure to be applied. Wastes containing less than 0.5 percent dry solid material are classified as liquid wastes and after filtration, are defined as the final TCLP extract. If the wastes contain greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Samples for volatiles analysis are extracted in a special pressurized extraction vessel. Extractions are conducted for a period of 18 ± 2 hours, followed by analysis of the extracts by approved EPA methodologies.

3.0 Definitions

TCLP: Toxicity Characteristic Leaching Procedure

Standard definitions can be found in Appendix 2 of the glossary in the Laboratory Quality Assurance Manual.

Percent Solids: That fraction of a waste sample from which no liquid may be forced out by an applied pressure.

4.0 Interferences

Potential interferences that may be encountered during analysis are discussed in individual analytical methods.

Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem. All glassware and equipment used for TCLPs destined for metals analysis must be rinsed with 12.5 percent nitric acid prior to washing to minimize metals contamination.

Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for metals.

5.0 Safety

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

5.1 Specific Safety Concerns or Requirements

The use of safety glasses and protective clothing are required throughout the entire procedure.

The solvents and reagents used in this extraction procedure are hazardous if improperly handled. Care must be taken during preparation and use of acetic acid, hydrochloric acid, nitric acid and sodium hydroxide solutions. Additional health and safety information is available and must be read from the Safety Data Sheets (SDS) maintained in the laboratory.

The acetic acid extraction fluid in the nonvolatile extraction vessels may react with carbamates in the sample to form CO₂ gas. Pressure buildup could potentially cause the vessels to explode. The vessels should be periodically vented during extraction, and once again prior to removal from the rotation apparatus to prevent this occurrence.

Proper precautions must be taken when using pressurized nitrogen during the filtration and pressurized procedures. All steps of procedure should be done under a fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Agitation apparatus: The rotation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

PH meter: calibrated daily

6.2 Supplies

Extraction Vessels:

Zero-Headspace Extraction Vessel (ZHE)-This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device and effectively precludes headspace. The vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110mm filter. The device contains O-rings, which should be replaced frequently.

Bottle Extraction Vessel- Borosilicate medium walled glass, Teflon screw cap, for semi-volatile and pesticides.

2 Liter plastic extraction bottles with lids, for metal only extractions.

TCLP-ZHE Filtration Apparatus.

ZHE Extraction Fluid Transfer Device

A pH Meter and pH probe-accurate to ± 0.05 units at 25° C. Prior to TCLP pH measurements, calibrate the pH meter and electrode in accordance with the manufacturer's recommendations. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

Laboratory Balance-balance must be accurate to within ± 0.01 grams. All weight measurements are to be within ± 0.1 grams.

Magnetic stirrer

Glassware:

- Beakers, glass 250mL
- Graduated cylinders, 2000mL.
- Erlenmeyer flasks, glass, 1000-mL.
- Whatman Glass Microfiber Filters – grade GF/F.
- Pre-washed Nitric filters purchased by ESS.
- Pre-preserved 40-mL vials.

Mortar and Pestle.

Standard Sieve 9.5mm.

Compressed Nitrogen

7.0 Reagents and Standards

Water for nonvolatile extractions is ASTM Type II from the deionized water system in the Organic Prep Lab.

Reagent Water for Volatile extractions is generated from the Volatile-Free purification system located in Organic Prep area.

Glacial Acetic Acid, ACS reagent grade.

Hydrochloric Acid (1N), ACS reagent grade.

Sodium hydroxide (10N), ACS reagent grade.

Nitric acid (1N), ACS reagent grade.

Extraction Fluid #1, pH=4.93 (Section 10).

Extraction Fluid #2, pH=2.88 (Section 10).

1.68 pH Buffer, 4.01 pH Buffer, 7.00 pH Buffer, 10.00 pH Buffer

8.0 Sample Collection, Preservation, Shipment and Storage

A minimum of 100g of sample is required for Organics/Metals. A minimum of 25g is required for VOA.

Preservatives shall not be added to samples prior to extraction.

When the waste is to be evaluated for volatiles analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in order to prevent the loss of volatiles analytes.

TCLP extractions and the analysis of the extracts must be conducted within the time period specified in Section 9. Extracts to be analyzed for metals must be preserved with 1N ACS reagent grade nitric acid to a pH of less than 2, unless precipitation occurs. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts for volatiles shall not be allowed to come into contact with the atmosphere (no headspace) to prevent losses.

9.0 Quality Control

9.1 Sample QC

Leachate Blanks

Leachate Blanks (LB) – One leachate blank must be extracted per batch of 20 samples and for each extraction fluid used. The leachate blanks are performed in the extraction vessels used for samples. The leachate blank must be demonstrated to be free of contamination.

The nonvolatile extraction vessels are 2-L borosilicate glass bottles with screw caps. A separate leachate blank is required for metal extractions done in plastic containers.

The volatile extraction vessels are ZHE units.

A minimum of one leachate blank (using the same extraction fluid as used for the samples) should be analyzed for every 20 extractions that have been conducted in an extraction vessel, provided that sample volume permits. If sample volume does not permit, sequential rotation of the extraction vessels used as leachate blanks should be employed. For example: For the vessel rotation, begin by using Extraction vessel #1 as the LB. Note the LIMs bench number in the TCLP rotation spreadsheet and note the use of vessel #1 in the LIMs bench sheet. For each subsequent batch and leachate blank, rotate through the vessels sequentially (ie. For the next batch use vessel #2, the next batch use vessel #3 and so forth). Once all extraction vessels have been used as a LB, start the rotation again using extraction vessel #1 and repeat the procedure. (See attachment #5)

Note: the department maintains a maximum of 20 extraction vessels in service at a given time. The number of vessels in use will be listed on the TCLP Leachate Blank Vessel Rotation Spreadsheet.

Using a 2000mL graduated cylinder add extraction fluid #1 (or extraction fluid #2) depending on preliminary sample evaluation, to the vessel. Place the container in the tumbler and secure the lid. Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours.

TCLP extractions, preparations and analyses must be conducted within the following time periods:

From Field Collection to TCLP Extraction (Filtration)

Volatiles	14 days
Semivolatiles	14 days
Mercury	28 days
Metals, except mercury	180 days

From TCLP Extraction to Preparation Extraction

Volatiles	Not Applicable
Semivolatiles	7 days
Mercury	Not Applicable
Metals, except mercury	Not Applicable

From Preparation Extraction to Analysis

Volatiles	14 days
Semivolatiles	40 days
Mercury	28 days
Metals, except mercury	180 days

Total Elapsed Time

Volatiles	28 days
Semivolatiles	61 days
Mercury	56 days
Metals, except mercury	360 days

9.2 Instrument QC N/A

10.0 Procedure

10.1 Sample Preparation

The TCLP preparation procedure can be broken down into four sections:

- Preliminary Sample Evaluation
- Preparation of Extraction Fluids
- TCLP Extraction Procedure for Nonvolatile Analytes
- TCLP Extraction Procedure for Volatile Compounds

10.1.1 Preliminary Sample Evaluation

A preliminary evaluation of the samples is performed prior to TCLP extraction. The results of the evaluation determine how the extraction is conducted and how the results are reported. The preliminary evaluation includes the following:

- Verification that sample contains no obvious liquid phase. Press down on sample with tongue depressor. If sample contains no obvious liquid, proceed to particle size reduction. If sample does yield liquid, determine percent solids
- Determination of percent solids using page 2 tab of spreadsheet.
- Determination if the waste contains insignificant amount of solid material, and is therefore the TCLP extract after filtration.
- Determination if the solid part of the waste needs particle size reduction.
- Determination of the extraction fluid to be used for the nonvolatile extractions, based on the pH of the waste.

10.1.2 Liquid Samples Extraction (Filter Only samples):

If the samples have been found to contain less than 0.5% dry solids, the filtrate is the TCLP extract.

Using a GF/F filter and filter apparatus collect the filtrate into the appropriate container. If metals are to be analyzed, acid treated filter papers are used.

Once filtered, samples are relinquished to the appropriate department.

Each filter only sample batch requires one leachate blank (LB) sample. DI H₂O is to be used as the extraction fluid for metals/extractable samples.

If the filtrate is to be analyzed for metals, the pH is adjusted by the Metals Department to less than 2 with 1N metals grade nitric acid. Check an aliquot for precipitation before acidifying the entire extract. If a precipitate does form, do not adjust the pH of the extract. Analyze the extract for metals as soon as possible.

The Extractable portion is stored at 4° C in main sample cooler.

10.1.3 Percent Solids Determination:

If samples are not 100% dry, the multi-phase spreadsheet must be used.

NOTE: If the sample is a mixed phase sample, contact the PM to determine which phase the client is interested in.

Use the following procedure in conjunction with tab 2 of the excel spreadsheet.

The samples are filtered under pressure through glass Microfiber filters. Prior to assembly of the filtration apparatus, clean all parts by washing with soapy water followed by rinsing with deionized water.

Follow all steps listed in the excel spreadsheet. Cells in green need to be filled in. Cells in orange are calculations or constants. Cells in yellow are informational.

Record weight of empty container used to collect the liquid phase from sample. Label container with job number and liquid phase. This volume is saved for recombination after tumbling.

You will be filtering the entire sample volume, so verify the analyses assigned, and that you will have enough volume.

Record the weight of the sample added to the pressure filtration unit.

Verify that the empty container is in place to collect the liquid that is being filtered out. Transfer sample to filtration unit, taking care to spread the sample layer evenly over the filter paper.

Gradually apply pressure until the liquid flow has ceased and pressurized gas moves through the filter. Do not exceed 50 psi.

Record the weight of the liquid filtered and the container in the spreadsheet. Record the pH reading of the liquid filtered.

The spreadsheet automatically calculates the % solids. The weight of the dry sample after pressure filtration gives an approximate weight of the amount of soil remaining on the filter paper. Use this information to determine if there is enough solid material to set the sample. (100 gram minimum for Metals and Extractables, 25 gram minimum for Volatile).

Continue with procedure as outlined in sections 10.1.4 -10.1.9.

After 18 +/- 2 hours of tumbling, record the tumbler end time and the min/max temperature readings. Remove samples from the tumbler. Test a small aliquot of the portion filtered before tumbling and the liquid from the tumbled portion for miscibility.

If the samples are incompatible, they can not be recombined and must be relinquished as individual samples.

If the samples are compatible, filter the tumbled portion with regards to the analysis assigned. (Metals samples are filtered through nitric rinsed filter paper, Extractable samples are filtered through unpreserved filter paper.)

Record the volume of the filtered sample. The spreadsheet automatically calculates how much liquid using pressure filtration to add to the liquid filtered from the tumbled portion.

10.1.4 Particle Size Reduction:

If the solid material in the sample can pass through a 9.5 mm sieve (less than 1 cm in diameter), particle size reduction is unnecessary.

If the samples need particle size reduction, crush or grind the sample with a mortar and pestle or whatever means necessary to reduce the sample's particle size. Record in spreadsheet.

10.1.5 Fluid Determination for Non volatile Analyses:

An aliquot of sample is initially tested for pH. The results determine which fluid is used for nonvolatile TCLP Extraction. Volatile TCLP extraction uses only extraction fluid #1 , prepared with volatile free reagent water.

Weigh out 5.0 g of representative sample. Record weight in spreadsheet.

Using a 100-mL graduated cylinder, add 96.5 ml of DiH₂O and stir the sample for 5 minutes using a magnetic stirrer.

Measure the pH of the sample using a calibrated pH meter and record in the spreadsheet. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

If pH is less than 5, use extraction fluid #1 for the TCLP sample extraction.

If pH is greater than 5, using a disposable 10mL pipette, add 3.5 ml of 1N HCl and swirl. Warm the sample on a hotplate to 50°C and hold at that temperature for 10 minutes.

Allow the solution to cool to room temperature, measure the pH, record in the logbook. If the pH is now less than 5, use extraction fluid #1. If the pH is still greater than 5, use extraction fluid #2.

10.1.6 Preparing Extraction Fluid:

If the extraction fluid is made more than 24 hours before use, the pH must be checked prior to extraction.

Care must be taken to ensure adequate mixing of large volumes to make certain that a stable pH has been reached prior to recording the result.

Preparation of Extraction Fluid #1 (pH 4.93 + 0.05)

Take 24L carboy of Fluid # 1 and take Di Water about 19L under the calibrated 20L mark. Add 114ml of Glacial acetic acid plus 128ml of 10N NaOH. Then make final volume 20L with Di water. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 ± 0.05 . Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet. Pre-made fluid concentrate, purchased from Environmental Express can also be used.

Preparation of Extraction Fluid #2 (pH 2.88 + 0.05)

Take 24L carboy of Fluid # 2. Take Di Water about 19L under the calibrated 20L mark. Add 130ml of Glacial acetic acid then make Final volume 20L by adding Di water and invert repeatedly to mix well. The pH of this fluid must be 2.88 ± 0.05 . Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet. Pre-made fluid concentrate, purchased from Environmental Express can also be used.

Preparation of the Volatile Free Extraction Fluid for ZHE (pH 4.93 + 0.05)

Take 24L carboy of the Volatile Free extraction fluid and take Volatile Free water 15L under the calibrated 16L mark. Add 90ml Glacial acetic acid and 102ml of 10N NaOH. Then make Final volume 16L by adding Volatile Free water. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 ± 0.05 . Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet.

10.1.7 TCLP Extraction Procedure- Nonvolatile Samples Metals/Extractables

This procedure describes the TCLP extraction of samples for Extractables (semivolatile, pesticide, herbicide) and metal analysis.

10.1.8 Glassware Preparation:

All extraction vessels, glassware and utensils used for the extraction procedure must be washed with soapy water, rinsed with dilute nitric acid, rinsed with tap water and then followed by a rinse with reagent grade water.

10.1.9 Setting Solid Phase Samples (TCLP Organics, TCLP Metals):

If the sample is found to be 100% solids, and requires no particle size reduction (or has undergone reduction).

Weigh out 100g sample and transfer to the extraction vessel. Record the weight and the vessel number in the spreadsheet.

The volume of the extraction fluid used is 20 times the sample weight (100g sample aliquot to 2000mL of extraction fluid). If there is insufficient volume the client must be notified before proceeding

After the sample and appropriate extraction fluid has been added to the vessel record the initial pH of extract in the spreadsheet. Cap the vessel and clamp the vessels to the rotator.

Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours. Record the analyst, date, time and temperature at the beginning and end of the extraction. The temperature in the room should be maintained at $23 \pm 2^\circ \text{C}$ and checked using a min/max thermometer. When the extractions are complete, remove the extractors from the rotator and separate the liquid and solid phases by filtering through a new glass fiber filter (GF/F).

Carefully decant the extraction fluid into the filtration apparatus. Collect the fluid in appropriate containers; glass for extractables and plastic for the metals. Discard the solid material left in the vessel.

Measure and record the final pH of the final extract.

If metals are to be analyzed, the filter must be acid washed and the pH of the extract corrected to < 2 with 1N metals reagent grade nitric acid.

Store the extractable portion in the AFSCEE sample cooler. Release the metals portion to the metals digestion analyst. Update status of the samples in the LIMS System.

10.1.10 Mixed Phase Samples:

If samples are mixed phase, use the Multi-phase tab on the TCLP spreadsheet.

For TCLP Metals/Extractables: Record weight of empty filtrate container (record in spreadsheet). Add 100 grams of mixed phase sample to filtration unit (weight of Wet + Dry sample in spreadsheet). Gradually increase pressure until all liquid is filtered from sample. Weigh Liquid + Container and record in spreadsheet. The spreadsheet will automatically calculate the % solids, weight of the dry sample, and the appropriate amount of extraction fluid to add. Tumble the solid portion for 18 ± 2 hours, store the liquid portion of the evaluation after the solid portion has tumbled. After tumbling test a small portion of both liquids for compatibility. If compatible, combine filtered liquid portion with tumbled portion. Filter entire volume following filtration procedures outlined in section 10.1.9. If incompatible, relinquish both portions to analytical departments for analysis.

For VOA TCLP: All filtration is to be done in the ZHE unit used to set the sample in.

Record weight of empty filtrate container (use a VOA vial to collect) in spreadsheet. Add 25 grams of mixed phase sample to filtration unit (weight of Wet + Dry sample in spreadsheet). Gradually increase pressure until all liquid is filtered from sample. Weigh Liquid + Container and record in spreadsheet. The spreadsheet will automatically calculate the % solids, weight of the dry sample, and the appropriate amount of extraction fluid to add. Tumble the solid portion for 18 ± 2 hours, store the liquid portion for evaluation after the solid portion has tumbled. After tumbling, test a small portion of both liquids for compatibility. Take care to minimize exposure of either portion to air to reduce loss of target analytes. If compatible, combine filtered portion with tumbled portion. If incompatible, relinquish both portions to analytical department for analysis.

10.1.11 Setting Solid Phase Samples (TCLP ZHE):

This method is used for the TCLP extraction of samples for volatile analysis. Do not allow waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done

when cold (4°C) to minimize loss of volatiles. Headspace should not be allowed in any of the extraction or collection containers.

NOTE: If the sample consists of pure oil, paint or solvent, consult the supervisor before proceeding. If the sample matrix is deemed unacceptable for the ZHE extractor, the supervisor is required to contact the Project Manager to notify the client. The project manager and client would then evaluate if a total Volatile analysis of the sample will meet the needs of the project. The analytical test would be updated to reflect a total analysis. The results of this 'Total Analysis' should be considered approximately 20 times higher than a TCLP leaching procedure.

Assemble the ZHEs:

All pieces of ZHE apparatus are labeled and assembled as a single unit. Assuming the vessel is unassembled, first place the Model #3745-ZHE body (3) with either end up and install the air side flange O-ring (12) in the gland on top of the body.

Replace the piston O-rings (12) on the piston (5) by stretching them over the piston and into the gland. NOTE: Be careful not to "roll" the O-rings into the gland. Wet the piston with extraction fluid.

Align the piston (5) carefully with the top of the body (3) and gently press it into the body. Continue pressing the piston into the body until it is completely inside the cylinder. NOTE: Care is necessary not to damage the O-rings. It may be helpful to moisten the O-rings with extraction fluid.

Locate the air side flange (1) and place it on top of the ZHE. Visually align the holes in the flange and the ZHE body and secure the flange with three knobs (8). Uniformly tighten the knobs.

Invert the partially assembly ZHE such that the air side flange (1) is now down.

Prepare a new Filter-Pak by placing a glass filter element between the two Stainless steel screens. Set the assembled filter-Pak to the side, until sample is added.

Locate the waste side flange (2) and install a Filter-Pak O-ring (12) in the inner gland of the flange. Install the body O-ring (12) in the outer gland of the waste side flange. NOTE: Be careful not to "roll" the O-rings into the glands.

Invert the waste side flange (2) and place it on top of the ZHE, aligning the holes at the same time.

Place the assembled ZHE with liquid inlet/outlet valve (14) on the top.

Verify that both the liquid inlet/outlet valve (13) and the quick-exhaust/relief valve (16) are open. NOTE: The quick exhaust/relief valve is open when the handle is up and the liquid inlet/outlet valve is open when the arrow points up.

Transfer of Sample to the ZHE

If the sample matrix is 100% solid material, and particle size reduction is unnecessary, weigh 25g of sample to the nearest 0.1g. Record the weight in the spreadsheet.

If particle size reduction is needed, reduce size, and then transfer 25g (+/- 0.1) to ZHE.

If the sample matrix is a mixed phase, use the percent solids information and the following calculation to determine the correct sample size to use:

$$\text{Wt of Waste to Charge (grams) ZHE} = \frac{\text{25 grams}}{\text{Percent Solids}} \times 100$$

Pour the appropriate weight of the mixed waste slurry into a tared beaker and transfer to the ZHE, quickly attaching the top flange. Reweigh the beaker and record in the weight ion the spreadsheet.

Introduce the sample to be extracted into the open top of the ZHE making sure that the piston (5) is far enough into the body (3) to provide sufficient free volume. Weigh out a representative sample of a maximum of 25.00 ± 0.1 g. Record the weight in the spreadsheet and the number of the vessel being used.

Use the following formula to determine how much extraction fluid to add to the ZHE:

$$\frac{\text{Wt of the extraction fluid} = 20 \times \text{percent solids} \times \text{Wt of waste filtered}}{100}$$

For example, if the sample has been classified as 100% solid, 500 mL of extraction fluid will have to be injected.

Check that the body O-ring (12) is properly seated in its gland and reinstall the Filter-Pak in the top of the ZHE. NOTE: Center the Filter-Pak carefully in its recess.

Verify that the Filter-Pak O-ring (12) and the waste side flange O-ring (12) are properly seated in their respective glands. Place the waste side flange (2) back on the unit and uniformly tighten the three knobs to close the vessel.

Close the quick-exhaust/ relief valve (16) on the air side flange (1). Do not close the liquid inlet/outlet valve. NOTE: The quick-exhaust/relief valve is closed when the handle is horizontal.

Introduce the proper amount of extraction fluid into the vessel using a peristaltic pump. The object is to put the 500 ml of extraction fluid into the vessel without opening it as to not expose the sample inside to the atmosphere. A metering pump is now set up next to the vessel.

First, with the tubing off of the top of the vessel, open the 2-way stainless steel valve and the vent relief valve on the bottom of the vessel. Now, start the metering pump (containing 500 ml of extraction fluid). When the fluid is almost to the end of the tubing, connect the tubing to the Luer fitting on top of the vessel securely. The fluid should start flowing into the vessel, pushing the piston back down to the bottom of the cylinder. The process may be slow. When all the fluid has been pushed into the vessel, shut the pump off, close the 2-way stainless steel valve, and close the vent relief valve on the bottom of the vessel. Disconnect the tubing and set the pump apparatus aside.

Close the liquid inlet/outlet valve (13) and the quick-exhaust/ relief valve (16). Close the valve on the syringe and disconnect it from the ZHE. Physically rotate the vessel end over end 2 or 3 times and place it back on the chair (6) with the liquid inlet/outlet valve on top.

Connect the pressure source to the air side flange and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) to bleed out any headspace that may have been introduced during the addition of extraction fluid and close the liquid inlet/outlet valve at the first sign of liquid. Slowly pressurize the ZHE unit to 50 psi. Allow the pressure gauge on the ZHE to stabilize and remove the pressure source. NOTE: A maximum of 10 psi is recommended for pressurization.

Place the ZHE in the rotary agitator (tumbler) and tumble for 18 ± 2 hours at 30 ± 2 RPM. The temperature in the room should be maintained at $23 \pm 2^\circ$ C. Record all information in the LIMs bench sheet.

When rotation is complete, allow the ZHE units to sit for 1 hour before filtering the leachate. Check that the ZHE unit has not lost pressure and record the final pressure reading in the LIMs bench sheet under Pressure_Final. If the vessel is no longer pressurized, perform maintenance on the unit and repeat the extraction with a new sample and different ZHE unit.

10.1.12 Filtering TCLP ZHE

Once the contents of the ZHE have settled, attach a pressure regulated source (set to 0 psi) of filtered compressed air or dry nitrogen to the gas inlet quick disconnect (14) found on the air-side flange (1)). Note: Compressed air must be prefiltered (5micron rating) to prevent particulate matter from scoring the walls of the ZHE.

Increase the supply pressure to 5-10 psi and note the pressure on the vessel gage (17) is approximately 2-5 psi. This indicates that the piston (5) is in motion and removing the sample headspace. At the first sign of liquid release from the liquid inlet/outlet valve (13) immediately close the valve (13). Disconnect the pressure source and open the quick-exhaust/relief valve (16) to depressurize the ZHE. NOTE: This will take a short period of time depending on the volume of free air remaining on the sample side of the vessel. NOTE: Pressure greater than 10 psi may be necessary.

Attach an appropriate filtrate collection container to the liquid inlet/outlet valve and firmly hand tighten the Speed-Nut (10). Close the quick-exhaust/Teflon syringes.

Reattach the pressure source to the vessel and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) on the ZHE. Gradually increase the supply pressure in 10 psi increments (up to the maximum of 50 psi) until no more initial liquid phase is expelled in a two minute interval or until the 40mL vial is full and free of any visible air bubbles. NOTE: The pressure gage (17) on the vessel should be allowed to stabilize at the line pressure setting before beginning the two-minute period.

Close the liquid inlet/outlet valve(s), disconnect the pressure source and remove the collection device. Depressurize the ZHE by slowly opening the quick-exhaust/relief valve (16). NOTE: Leave the quick-exhaust/relief valve open.

10.1.13 Cleaning and Maintaining the ZHEs

Before the ZHEs are assembled, wash all the parts with hot, soapy water followed by rinsing with tap and deionized water. A sonic bath can be used to clean the more difficult parts. After cleaning, the cylinder, piston, stainless steel screens, and any rubber O-rings

can be placed in the 103°C oven for a few hours. Only these parts can be heat treated as the heat damages any part containing a valve. Make sure the parts are room temperature before setting up samples. Make sure the pistons do not get interchanged. ZHE O-rings must be free of cuts or cracks or the extractors may leak. Before assembly, examine the O-rings for damage.

10.1.14 Aqueous Samples

If the samples have been found to contain less than 0.5 percent dry solids, the filtrate is the TCLP extract.

Collect the filtrate directly into a 40 mL VOA vial pre-preserved with HCL, allowing for no headspace to form.

Each filter only batch must contain one leachate blank (LB) sample. VOA Free H₂O is to be used as the extraction fluid for VOA samples.

Store the vials in the GC/MS sample cooler. Release samples in the LIMS System.

10.2 Calibration

The pH meter is calibrated every day prior to use. The calibration is performed with a 2.01 pH buffer solution, a 4.01 pH buffer solution, a 7.01 pH buffer solution and a 10.01 pH buffer solution and then checked against a 7.01 pH buffer solution from a different vendor as a second source. The check must be accurate within ± 0.05 . If the check fails, repeat the calibration until a passing check has been achieved. Calibration slope and pH of the second source is recorded in the TALS bench sheet every day.

Because pH buffers are used to calibrate and verify the pH meter, they must be logged into the LIMS database and given an individual tracking number. This LIMS generated tracking number will be recorded in the appropriate section on the spreadsheet.

Analytical balances are calibrated every 12 months and checked daily to ensure calibration is maintained.

11.0 Calculation/ Data Reduction

See calculations noted in Procedure.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

Refer to the laboratory QA manual for specific demonstration of capabilities procedures.

12.3 Training Requirements

Refer to the laboratory QA manual for specific training requirements.

13.0 Pollution Control

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

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. . All waste will be disposed of in accordance with Federal, State and Local regulations. Waste disposal procedures are incorporated by referencing section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

The following waste streams are produced when this method is carried out:

Acidic waste from sample extract: All acid waste is disposed of in "A" waste satellite containers (except nitric acid waste). When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

Solid waste from sample extract: The solid waste from the sample extract is dried and disposed of in a "BE" satellite container. When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

Remaining TCLP extracts: The remaining TCLP extracts are considered "A" waste and are disposed of directly to a 55-gallon "A" waste drum by appropriately trained laboratory technicians in accordance to all state and federal regulations.

15.0 References / Cross-References

Method 1311, "Test Methods for Evaluating Solid Waste", EPA SW846 Third Edition, 12/96

16.0 Method Modifications: N/A

17.0 Attachments

Figure 1: Tumbler- Rotary Agitation Apparatus

Figure 2: Zero Headspace Extractor (ZHE)

Figure 3: ZHE Specifications

Figure 4: ZHE and Fluid Metering Pump

Figure 5: Flow chart

Figure 6: Regulated analytes for Toxicity Characteristic with Regulatory Levels.

Attachment 1: Data Entry/Second Level Review for 1311/1312

Attachment 2: Multi-Phase Template

Attachment 3: TCLP Leachate Blank Vessel Rotation Spreadsheet

18.0 Revision History

Revision 7, dated 4 May 2016

- Changes made in Section # 10.1.6 for the preparation of TCLP extraction fluid # 1 , 2 and Volatile Free Fluid.

Revision 6, dated 2 December 2015

- Updated Section 10.1.11 to include specifications about sample handling procedures.
- Updated Section 10.2 to include specifications on pH meter calibration.
- Changed Lab Director and Department Manager, signatures added.

Revision 5, dated 29 October 2014

- Updated Section 9.1 to include the procedure for sequential rotation of the extraction vessels when used as leachate blanks.
- Updated Section 17.0 to include the TCLP vessel rotation spreadsheet and removed outdate spreadsheets

Revision 4, dated 28 August 2013

- Updated Section 10.1.11 to include pressurization of ZHE units to 50psi, to include documenting the pressure of the ZHE in the LIMs bench sheet and to include allowing the vessels to sit after rotation to perform pressure inspection.

Revision 3, dated 13 December 2012

- Removed section 10.1.13, Leak-testing the ZHE, as the procedure is no longer in practice at TestAmerica Buffalo.
- Changed QA Officer-signature changed.

Revision 2, dated 31 May 2011

- Added reagents 1.68 pH Buffer, 4.01 pH Buffer, 7.00 Buffer and 10.00 Buffer to Section 7.0.
- Updated Section 10.1.3 with current procedure for determining Percent Solids.
- Updated Section 10.1.10 with current procedure for handling Mixed Phase Samples for TCLP Metals and TCLP VOA samples.
- Added the following wording to Section 10.2, "Because pH buffers are used to calibrate and verify the pH meter, they must be logged into the LIMS database and given an individual tracking number. This LIMS generated tracking number will be recorded in the appropriate section on the spreadsheet".
- Updated Attachments 1-4.

- Added Attachment 5, "Data Entry/Second Level Review for 1311/1312".
- Changed Quality Manager, signature added.
- Changed Department Manager, signature added.

Revision 1, dated 13 March 2009

- Integration for TestAmerica operations
- Changed SOP references from logbook to spreadsheet
- Updated attachments to include spreadsheets

Figure 1. Tumbler- Rotary Agitation Apparatus

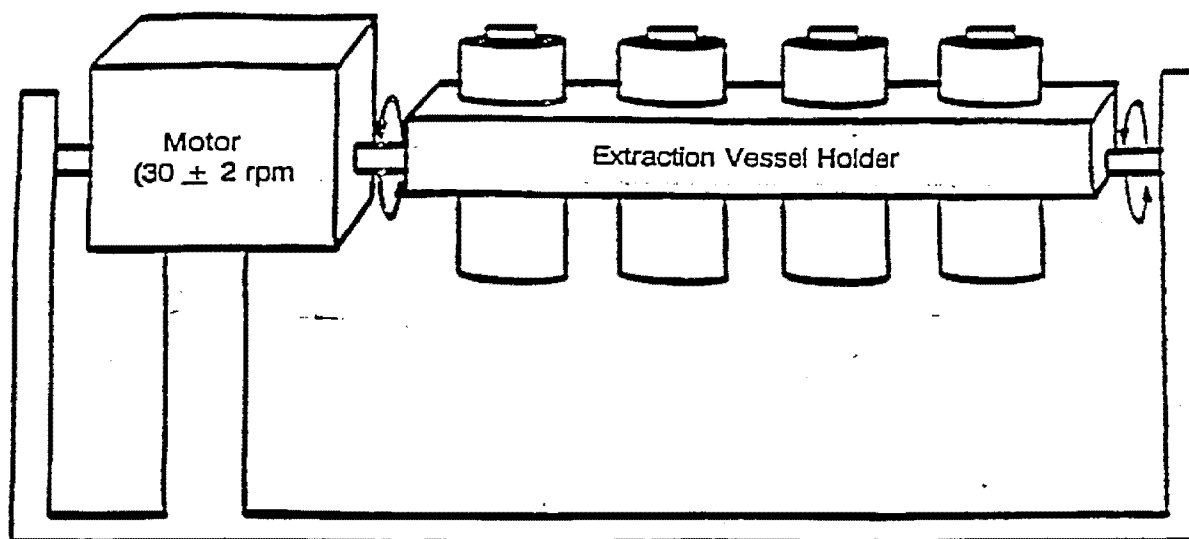


Figure 2. Zero Headspace Extractor (ZHE)

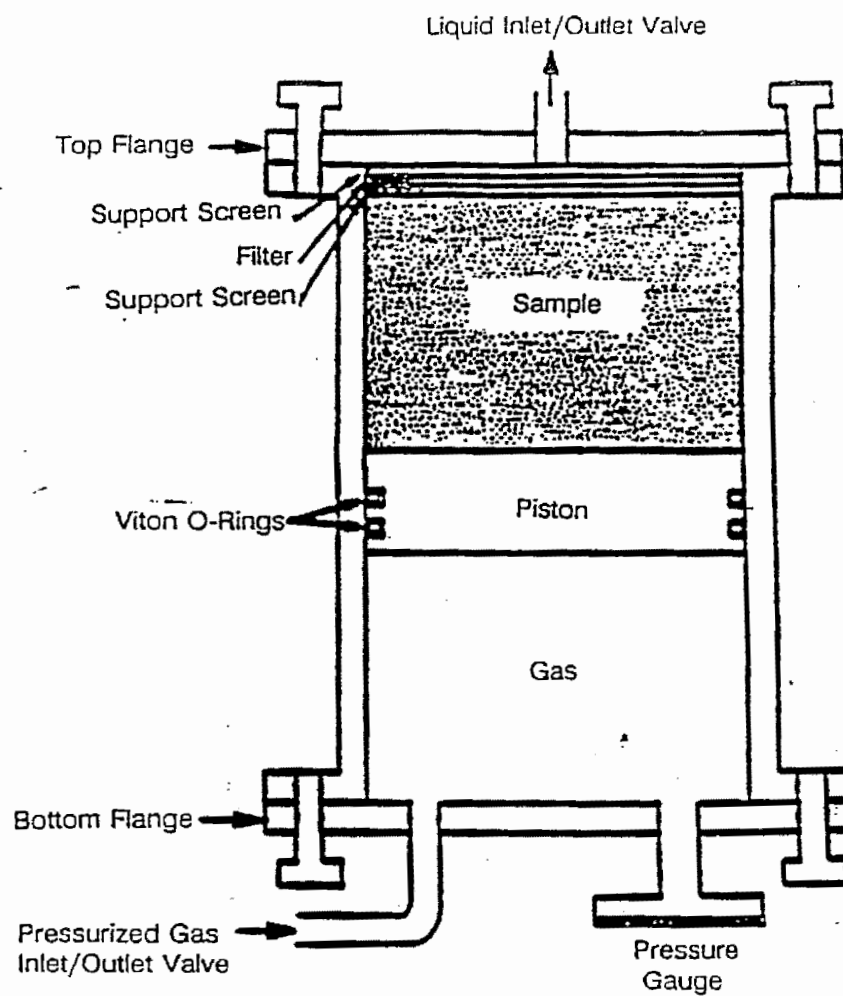


Figure 3: ZHE Specifications

MODEL 3745-ZHE SPECIFICATIONS

MATERIAL: 316 Stainless Steel, all welds inspected per MIL-I-6866. Standard Viton® O-Rings, supplied with an agitator collar, and a polypropylene chair for set-up.
DIMENSIONS: 127.00mm (5.00") dia., 241.30mm (9.50") plus sample fitting. Will easily fit Associated Design's ROTARY AGITATOR MODEL: 3740-4-BRE, 3740-6-BRE, 3740-8-BRE, and 374-12-BRE.
CONNECTIONS: Sample side supplied with 316 stainless Steel Luer-Lok® adapter and a Teflon® bushing adapter to accommodate Tedlar® bags. Airside supplied with stainless steel Swagelok® quick-connect fittings (double end shut-off) Viton® O-Rings, and stainless steel safety relief/quick exhaust valve

PRESSURE: Working pressure 50 psi
FILTER/PREFILTER SIZE: 90mm
.7 micron GF/F Filter
EFFECTIVE FILTRATION AREA: 45.8cm²
PISTON BREAKWAY FORCE: 2-5 psi
VOLUME: 550ml (nominal)
GAUGE: 2-1/2" dia., 0-60 psi glycerin filled

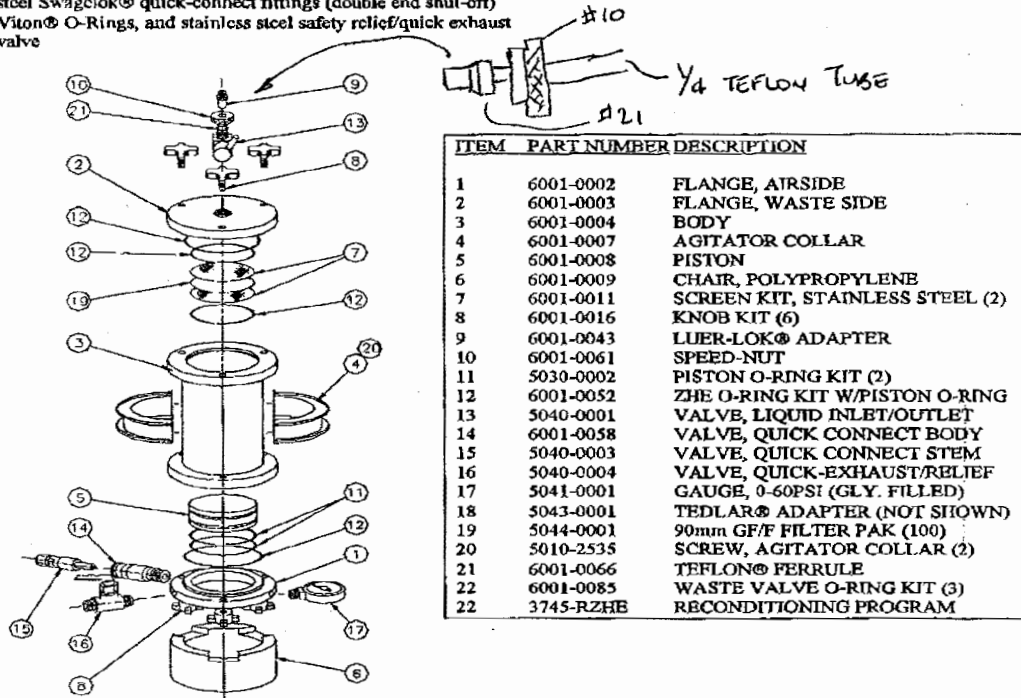


Figure 4. ZHE and Fluid Metering Pump

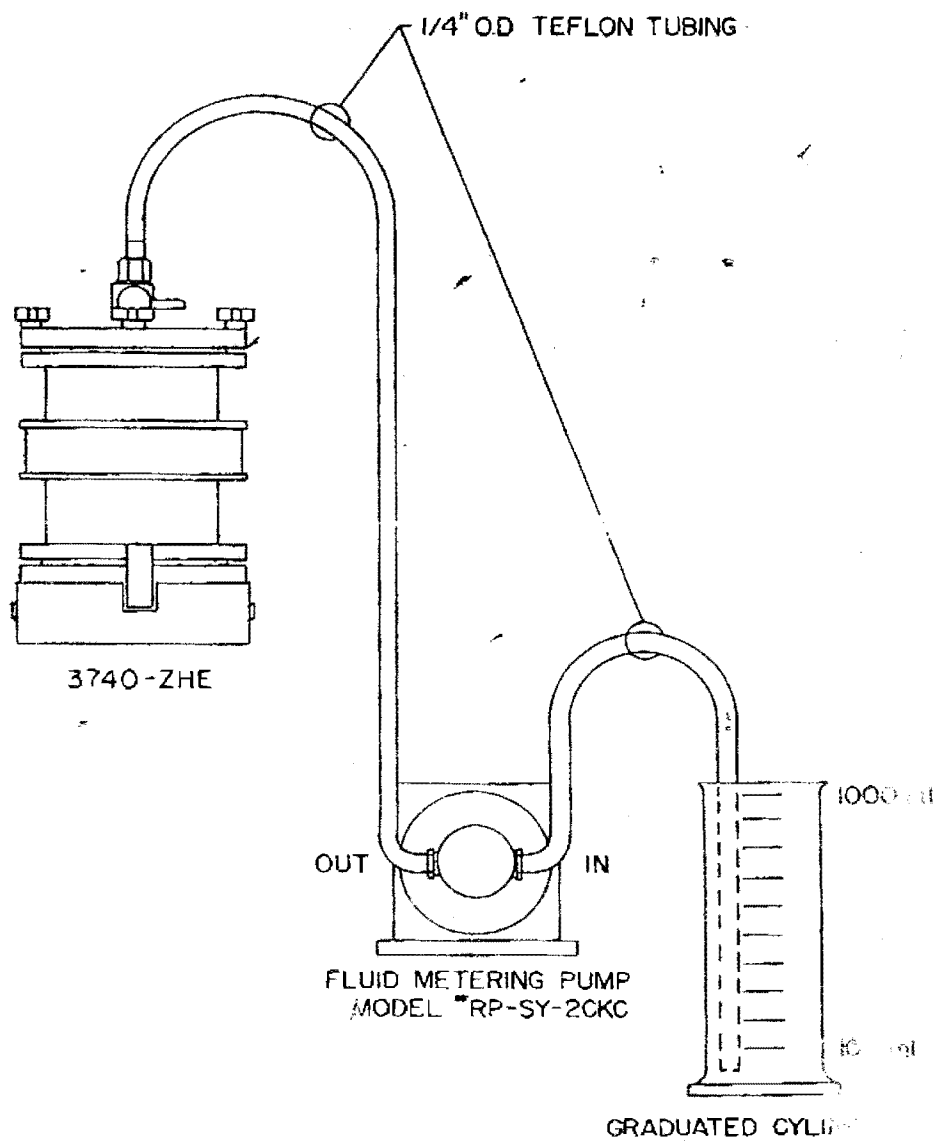


Figure 5. Flow Chart (page 1 of 2)

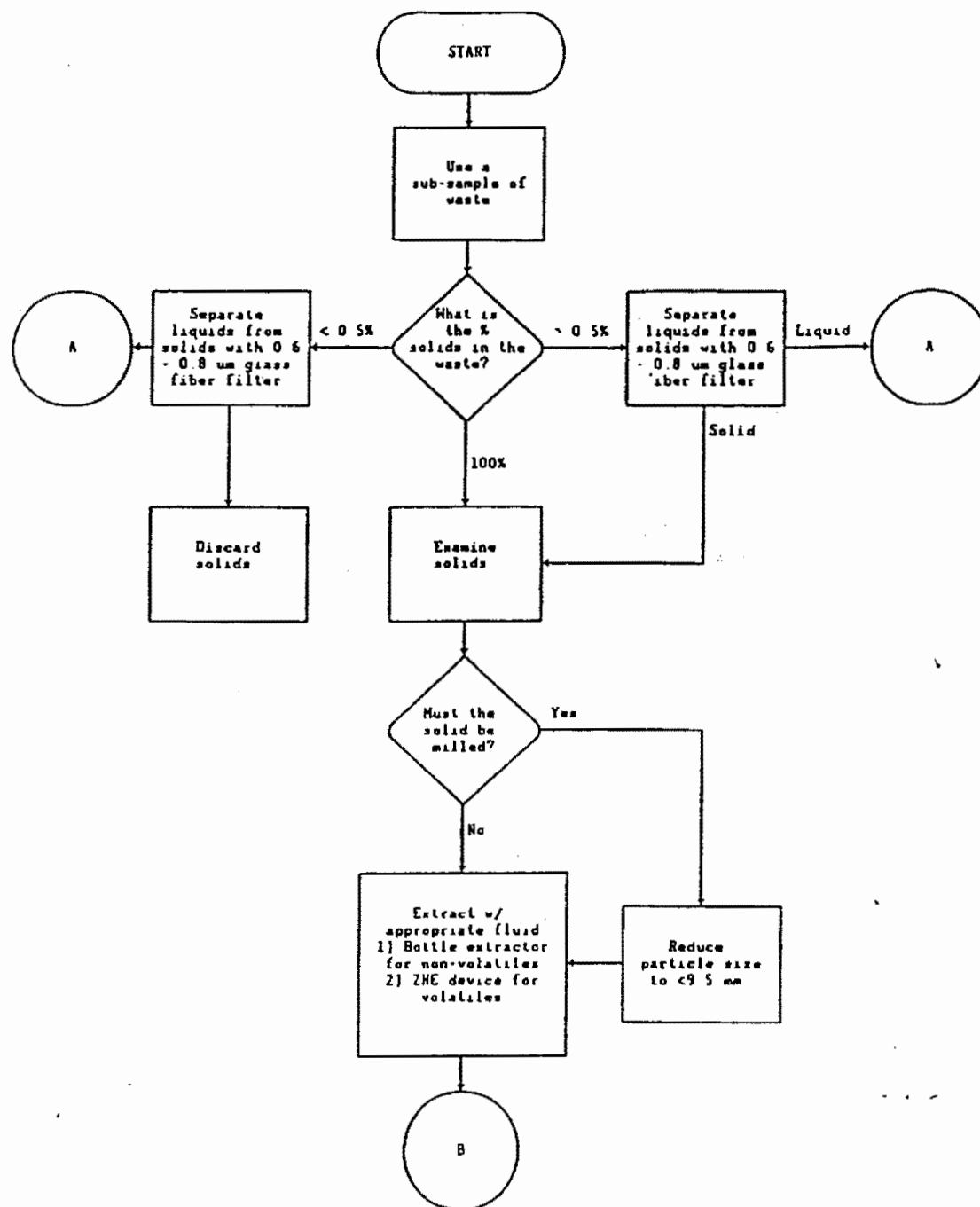


Figure 5. Flow Chart (page 2 of 2)

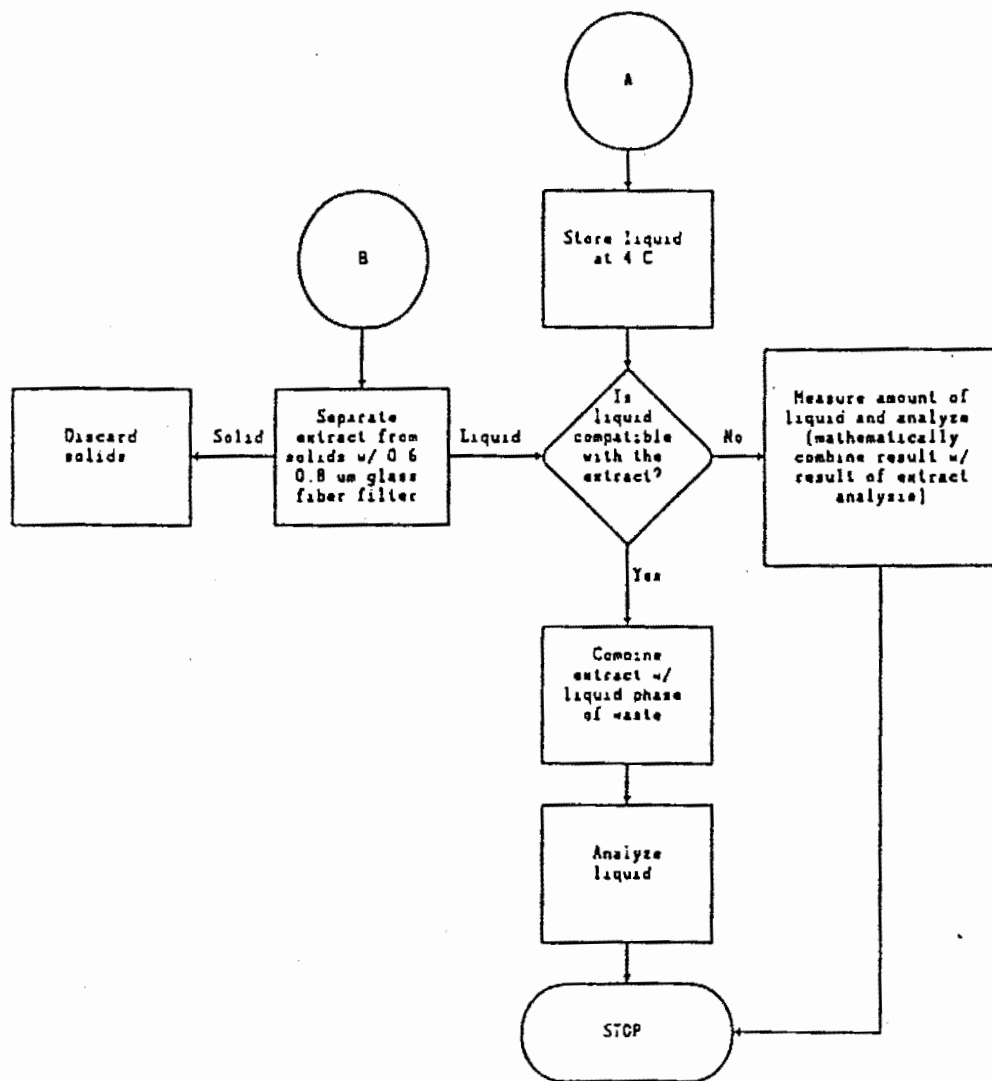


Figure 6: Regulated analytes for Toxicity Characteristic with Regulatory Levels.

<u>Compound</u>	<u>Regulatory Level (mg/L)</u>
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
2-Butanone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl chloride	0.2

9.2 Semivolatiles

<u>Compound</u>	<u>Regulatory Level (mg/L)</u>
2-Methylphenol	200.0
3-Methylphenol	200.0
4-Methylphenol	200.0
1,4-Dichlorobenzene	7.5
2,4-Dinitrotoluene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0

9.3 Pesticides

<u>Compound</u>	<u>Regulatory Level (mg/L)</u>
Chlordane	0.03
2,4-D	10.0
Endrin	0.02
Heptachlor	0.008
Heptachlor epoxide	0.008
Lindane (gamma BHC)	0.4
Methoxychlor	10.0
Toxaphene	0.5
2,4,5-TP (Silvex)	1.0

9.4 Metals

<u>Element</u>	<u>Regulatory Level (mg/L)</u>
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

Figure 6. Regulated Analytes for Toxicity Characteristic with Regulatory Levels

Attachment 1: Data Entry/Second Level Review for 1311/1312

Data Entry/Second Level Review for 1311/1312

- Specific Spreadsheets have been created for each unique Leaching procedure.
- Spreadsheets have been color coded: any green boxes require information to be filled entered, yellow and white boxes are for reference only.
- Data Entry (Set): refers to individual starting the leaching process.
- Data Entry (filter): refers to individual filtering the leached samples
- 2nd Level Review: refers to individual performing review of LIMS batches and spreadsheets to ensure that all required information is recorded, and method requirements have been met.

Metals/Extractables (not multiphase):

Data Entry (Set)

- Calibration/Verification of pH meter: record pH reading and LIMS ID. pH readings must be +/-0.05 of actual value.
- Record Job #, Extractions type, Vessel if applicable
- Record whether the sample needed particle size reduction (Y or N)
- Record weight of the test sample, and pH of test samples (fluid determining step)/or if sample is from East of the Mississippi or West of the Mississippi for SPLP extraction
- Record weight of the sample tumbled (If less than 100 grams a job exception must be sent to PM. If setting with >100 grams, appropriate qualifier, comment must be made)
- Record pH and LIMS ID of extraction fluid used. pH must be within method required range.
- Record initial pH of extract
- Record tumbler start time
- Ensure tumbler calibration is up-to-date and recorded (calibrate weekly)
- Create appropriate batches (by fluid type) and record batch# on spreadsheet
- Date and initial under Entry (Set)

Data Entry (Filter)

- Record stop time of tumbler
- Record Date and Time of filtration
- Ensure that temperature was maintained within required range (21-25) on min/max thermometer. If min/max readings are outside of range relinquish continual read thermometer to appropriate personnel for closer monitoring. Continual read will be used to indicate when temperature went outside of range (was it during the "tumble time", as well as if there was any impact on client data, specifically if the temperature was outside of the range for greater than 2 hours)
- Record Measured temperatures and Corrected temperatures (after correction factor applied)
- Record the volume of sample that was filtered
- Record final pH of extract prior to filtering
- Update LIMS system with filtration information (Element: Prepared = filtration date, Prepared By = person who did the filtering)

2nd Level Review

- Temperature was within required range, and recorded on spreadsheet
- Dates, times, and initials for setting and filtering have been recorded in spreadsheet
- Tumbler Calibration Date has been recorded and is up-to-date. If calibration of tumbler is needed, inform personnel performing "set"
- pH readings are within range (+/-0.05) and LIMS ID recorded on spreadsheet
- extraction fluid is within pH range and LIMS ID is recorded on spreadsheet

- All information regarding the leach procedure is recorded on spreadsheet including weights/volumes/pH readings
- If >100 grams used for sample, appropriate comment/qualifier was made in LIMS system.

Metals/Extractables (multiphase):

Data Entry (Set)

- Calibration/Verification of pH meter: record pH reading and LIMS ID. pH readings must be +/-0.05 of actual value.
- Record Job #, Extractions type, Vessel if applicable
- Record weights for empty container, weight and dry sample added, and filtered liquid/container.
- Record pH of liquid from pressure filtration
- Record weight of the test sample, and pH of test samples (fluid determining step)
- Record weight of the sample tumbled (If less than 100 grams a job exception must be sent to PM. If setting with >100 grams, appropriate qualifier, comment must be made)
- Record pH and LIMS ID of extraction fluid used. pH must be within method required range.
- Record initial pH of extract prior to filtering
- Record tumbler start time
- Ensure tumbler calibration is up-to-date and recorded (calibrate weekly)
- Create appropriate batches (by fluid type) and record batch# on spreadsheet
- Date and initial under Entry (Set)

Data Entry (Filter)

- Record stop time of tumbler
- Record Date and Time of filtration
- Ensure that temperature was maintained within required range (21-25) on min/max thermometer. If min/max readings are outside of range relinquish continual read thermometer to appropriate personnel for closer monitoring. Continual read will be used to indicate when temperature went outside of range (was it during the "tumble time", as well as if there was any impact on client data specifically if the temperature was outside of the range for greater than 2 hours)
- Record Measured temperatures and Corrected temperatures (after correction factor applied)
- Record whether liquid from pressure filtration is compatible with liquid that was tumbled. If liquids are compatible, filter leached portion, and combine with liquid from pressure filtration in the correct ratio.
- Record the volume of sample that was filtered
- Record final pH of extract
- Update LIMS system with filtration information (Element: Prepared = filtration date, Prepared By = person who did the filtering)

2nd Level Review

- Temperature was within required range, and recorded on spreadsheet
- Dates, times, and initials for setting and filtering have been recorded in spreadsheet
- Tumbler Calibration Date has been recorded and is up-to-date. If calibration of tumbler is needed, inform personnel performing "set"
- pH readings are within range (+/-0.05) and LIMS ID recorded on spreadsheet
- extraction fluid is within pH range and LIMS ID is recorded on spreadsheet
- All information regarding the leach procedure is recorded on spreadsheet including weights/volumes/pH readings
- If >100 grams used for sample, appropriate comment/qualifier was made in LIMS system.

ZHE (not multiphase):

Data Entry (Set)

- Calibration/Verification of pH meter: record pH reading and LIMS ID. pH readings must be +/-0.05 of actual value.
- Record Job # and Vessel
- Record weight of the sample tumbled (If less than 25 grams a job exception must be sent to PM. If setting with >25 grams, appropriate qualifier, comment must be made)
- Record pH and LIMS ID of extraction fluid used. pH must be within method required range.

- Record initial ZHE pressure
- Record tumbler start time
- Ensure tumbler calibration is up-to-date and recorded (calibrate weekly)
- Create appropriate batches and record batch# on spreadsheet
- Date and initial under Entry (Set)

Data Entry (Filter)

- Record stop time of tumbler
- Record Date and Time of filtration
- Ensure that temperature was maintained within required range (21-25) on min/max thermometer. If min/max readings are outside of range relinquish continual read thermometer to appropriate personnel for closer monitoring. Continual read will be used to indicate when temperature went outside of range (was it during the "tumble time", as well as if there was any impact on client data specifically if the temperature was outside of the range for greater than 2 hours)
- Record Measured temperatures and Corrected temperatures (after correction factor applied)
- Record Final ZHE pressure. If final pressure < initial pressure, sample must be re-set and zhe unit checked for leaks.
- Record the volume of sample that was filtered
- Update LIMS system with filtration information (Element: Prepared = filtration date, Prepared By = person who did the filtering)

2nd Level Review

- Temperature was within required range, and recorded on spreadsheet
- Dates, times, and initials for setting and filtering have been recorded in spreadsheet
- Tumbler Calibration Date has been recorded and is up-to-date. If calibration of tumbler is needed, inform personnel performing "set"
- pH readings are within range (+/-0.05) and LIMS ID recorded on spreadsheet
- extraction fluid is within pH range and LIMS ID is recorded on spreadsheet
- All information regarding the leach procedure is recorded on spreadsheet including weights/volumes/pH readings
- If >25 grams used for sample, appropriate comment/qualifier was made in LIMS system.
- If final ZHE pressure < Initial ZHE pressure, sample was removed from batch and re-set.

TCLP Multi-Phase Template
BF-OP-MP-007
Rev. 011/8/2014

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Attachment 3: TCLP Leachate Blank Vessel Rotation Spreadsheet

TCLP Leachate Blank Rotation Spreadsheet

BF-OP-TCLPBLK-006
Rev. 0 10/27/2014

	Batch Number																			
	12345																			
ZHE Extraction Vessel ID	1	LB																		
	2	X	LB																	
	3	X		LB																
	4	X			LB															
	5	X				LB														
	6	X					LB													
	7	X						LB												
	8	X							LB											
	9									LB										
	10										LB									
	11											LB								
	12												LB							
	13													LB						
	14														LB					
	15															LB				
	16																LB			
	17																	LB		
	18																		LB	
	19																			LB
	20																			LB

X - denotes vessel used for sample leachate

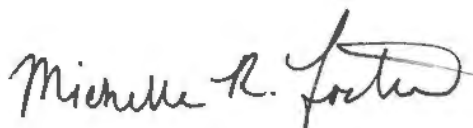
LB - denotes vessel used for Leachate Blank

Instructions: Perform sequential rotation of the extraction vessels used as a LB by batch. Starting with Vessel #1 as a LB, note the Batch # that the vessel was used for in the spreadsheet. For the next Batch #, use Vessel #2 and so on and so forth until all vessels have been used as a LB. Then begin again with Vessel #1 and repeat the procedure. Also, note which Vessel # was used for client samples by placing an "X" in the corresponding column.

Title: Total Cyanide
Methods 335.4, 9012A / 9012B, SM4500-Cn E, Lachat 10-204-00-1

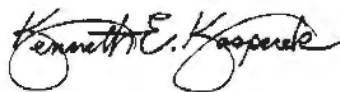
Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):



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Department Manager

8/13/15
Date



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8/13/15
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Date

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Applicable matrices are drinking, surface, saline waters, sediments and other solids including oils, domestic and industrial.

The laboratory standard quantitation limit is (0.01 mg/L) for aqueous samples and 1.0 mg/kg for soils.

2.0 Summary of Method

2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then analyzed colorimetrically.

2.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

3.0 Definitions

3.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

4.0 Interferences

4.1 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid which will react with some organic compounds to form oximes. These compounds formed will decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment of all samples and standards with sulfamic acid.

4.2 Sulfides adversely affect the colorimetric procedure. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce hydrogen sulfide during the distillation should be eliminated.

4.2.1 Steps to remove sulfide interference should only be used when the potential interferences are confirmed to be present since the treatment can affect cyanide recoveries.

4.3 The presence of surfactants may cause samples to foam during refluxing. If this occurs, the addition of an agent such as DOW CORNING 544 antifoam agent will prevent the foam from collecting in the condenser tube.

4.4 Oxidizing agents, such as chlorine, decompose most cyanide containing samples. Therefore, each sample is tested for chlorine using potassium iodide starch test papers

4.5 Thiocyanate is reported to be an interference when present at very high levels.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material ¹	Hazards	Exposure Limit ²	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2-ppm, 5 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Chloramine-T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.

Pyridine	Flammable Irritant	5 ppm- TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.3** All manipulations must be done in the hood so that any HCN gas that might escape is safely vented.
- 5.4** Many of the reagents used in these test methods are highly toxic. These reagents and their solutions must be disposed of properly.
- 5.5** The distilling unit should be set up and operated in a chemical fume hood suitable for use with caustic and corrosive materials. Hood face velocity should not be less than 100 CFM.
- 5.6** Wear hand and eye protection suitable for use with caustic and corrosive materials.
- 5.7** Hot surfaces during operation – do not touch.
- 5.7.1** Heat block operates at 125°C.
- 5.7.2** Exterior case near heat block may exceed 80°C.
- 5.8** Do not move unit while hot; sudden movement may result in bumping and sample loss.
- 5.9** Allow system to cool before removing glassware, separation of hot glassware components may result in bumping, boil-over and spray of hot corrosive materials.
- 5.10** Avoid breathing vapors, may be harmful or fatal. Review all safety data sheets for all materials utilized or generated in this operation.

5.11 Unplug unit prior to cleaning exterior surfaces of distilling unit (SHOCK HAZARD). Wipe with damp sponge or towel first with mild sodium bicarbonate or similar solution, followed by DI water, after each use.

5.12 The use of boiling chips is important when distilling samples.

5.13 THIS LIST CONTAINS SOME BASIC RECOMMENDED SAFETY PRECAUTIONS. IT SHOULD NOT BE CONSIDERED AS A COMPLETE OR EXHAUSTIVE LIST. MORE RIGOROUS OR ENHANCED PRECAUTIONS MAY BE NECESSARY WHILE USING THIS EQUIPMENT. PLEASE CONSULT YOUR SAFETY COORDINATOR AND SDS PRIOR TO USING THIS EQUIPMENT.

6.0 Equipment and Supplies

6.1 Instrumentation

- Reflux-distillation apparatus: Enviro Midi-Distillation system. Includes heat block with a variable timer, built in cold finger condensers with flowmeter and Teflon valves for controlling the vacuum for the scrubbers.
- Analytical balance
- Lachat Quikchem 8000: flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.
 - Sampler
 - Multi-channel proportioning pump
 - Reaction unit or manifold
 - Colorimetric detector (570nm) (similar to a spectrophotometer)
 - Data system

6.2 Supplies

- 20 glass tubes
- 10 absorber impingers with fitted ends
- 10 reflux impingers
- Culture tube racks
- 125 mL Erlenmeyer flasks
- Filter pump
- Miscellaneous laboratory glassware (volumetric flasks, graduated cylinders, etc.).
- Eppendorf pipettes
- Lead acetate test paper
- Potassium iodide – starch test paper

7.0 Reagents and Standards

7.1 VWR 10 N Sodium Hydroxide solution

- 7.2 VWR 0.25 N Sodium Hydroxide solution
- 7.3 VWR Magnesium Chloride 51% W/V solution
- 7.4 Sulfamic Acid
 - 7.4.1 50.0 g Sulfamic Acid to 500 ml DiH₂O.
- 7.5 50% Sulfuric Acid
 - 7.5.1 250mL deionized H₂O + 250mL Concentrated sulfuric acid
- 7.6 Cadmium Carbonate (Powder)
- 7.7 Ascorbic acid
- 7.8 ERA Soil in cyanide standard
- 7.9 Methyl red indicator
 - 7.9.1 Dissolve 0.1g methyl red sodium salt into 100 mL of Deionized H₂O.
- 7.10 Stock cyanide solution 1mL = 1 mg CN
 - 7.10.1 Purchased pre-made, two separate sources (Complex and Free.)
- 7.11 Phosphate Buffer, 0.71m
 - 7.11.1 Dissolve 97.0g potassium phosphate, monobasic, anhydrous into 1L of deionized H₂O. Refrigerate and store in dark bottle. Prepare biannually.
- 7.12 Chloramine-T solution
 - 7.12.1 Dissolve 8.0g Chloramine-T into 1-L of deionized H₂O. Refrigerate and store in dark bottle. Prepare daily.
- 7.13 Pyridine-Barbituric acid reagent
 - 7.13.1 In a fume hood place 15g of Barbituric acid into a 1L volumetric flask, and add just enough deionized H₂O to wash the sides of the flask and wet the barbituric acid. Add 75 mLs of pyridine while stirring until barbituric acid dissolves. Add 15 mLs of concentrated HCl, mix, and cool to room temperature. Dilute to 1L with deionized H₂O and mix. Refrigerate and store in dark bottle. Prepare weekly.
- 7.14 Intermediate Cyanide STD: 50ppm
 - 7.14.1 2.5mL of the 1000ppm Complex Cyanide Standard is added to 50mL deionized H₂O; this is used for digestion. Expires within two weeks.
 - 7.14.2 2.5mL of the 1000ppm Free Cyanide standard is added to 50mL deionized h₂O; this is used for the instrument CCV. Expires within one week.
- 7.15 Instrument Continuing Calibration Verification (CCV) Check: 0.25ppm
 - 7.15.1 0.25mL of the 50ppm Intermediate Cyanide Standard (7.14.2) is added to 50mL of 0.25N Sodium Hydroxide.

- 7.16** Calcium Hypochlorite: Dissolve 5g $\text{Ca}(\text{OCl})_2$ solution in 100mL deionized water. Store in an amber glass bottle in the dark. Prepare monthly.
- 7.17** Zinc Acetate Solution: Dissolve 120g $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ in 500mL deionized water. Dilute to 1L.
- 7.18** Sodium Acetate Buffer: Dissolve 410g sodium acetate trihydrate in 500mL deionized water. Add glacial acetic acid to bring pH of solution to 4.5 (approximately 500mL.)
- 7.19** Laboratory Control Standard (LCS): First digested batch needs a high-range check (0.4mg/L), a mid-range check (0.25mg/L) and a low-range check (0.1mg/L); all to be labeled LCSs. Each batch proceeding within the same business day should have a mid-range check (0.25mg/L) and a low-range check (0.1mg/L); both to be labeled at LCSs.
- 7.19.1** 0.4mL, 0.25mL, and 0.1mL of the 1000ppm Complex Cyanide Standard is added to 50mL of deionized water. This is then carried through the digestion process as samples are.
- 7.19.2** MCP requires an LCSD be digested with each batch of affected samples. In this case a duplicate LCS will also be digested. The LCS/LCSD should be digested at 0.25mg/L.
- 7.19.3** Prussian Blue : Purchased Iron(III) hexacyanoferrate III
- 7.19.4** Prussian Blue Intermediate Standard: Measure out 0.02g of Prussian Blue into a 500mL flask. Dilute to the line with DIH_2O and agitate to dissolve. This solution will be used for the LCSN for PAC requirements. Expiration: 3 Years.
- 7.19.5** PAC Buffer: Use a 2L Erlenmeyer flask for the preparation of this reagent. Add 500g of sodium dihydrogen phosphate monohydrate to 500mL of deionized water. Place the electrode of a calibrated pH meter into the solution and add concentrated phosphoric acid until the pH of the solution is 1.25. Start by adding 25mL portions of concentrated phosphoric acid until the pH falls to 2 and then continue using dropwise addition until the pH of 1.25 is reached. Expiration : 1 Year
- 7.19.6** Soil LCS uses a purchased Cyanide in soil ERA standard; 0.5g of ERA standard up to 50mL of deionized water. May need to be diluted on analytical side to get standard within curve range.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	Glass or Plastic	200mL	NaOH, pH >12; Cool 4 ± 2°C	14 Days	40 CFR Part 136.3 SM 4500-Cn ⁻
Soils	Glass or Plastic	50g	Cool 4 ± 2°C	14 Days	N/A

¹ Inclusive of digestion and analysis.

8.1 The sample should be collected in plastic or glass bottles 125mL or larger size. All bottles must be thoroughly cleansed and rinsed to remove soluble material. TestAmerica Buffalo purchases pre-cleaned bottles that come with a certificate of analysis.

8.2 Samples must be preserved with 2 mLs of 10 N NaOH per Liter of sample (pH>12) at the time of collection.

8.3 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator/cooler to maintain temperature at 4±2° C. Distillation and analysis for cyanide must be completed within 14 days after sample collection for methods 9012 and 335.4.

9.0 Quality Control

9.1 All QC samples must be in the established acceptable range set by Test America Laboratories.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS/LCSD)*	1 in 20 or fewer samples	+/- 10%
Soil Laboratory Control Sample (LCS)	1 in 20 or fewer samples	Statistical limits based off of ERA certified value
Continuing Calibration Blank (CCB)	1 in 10 or fewer samples	< Rpt. Limit
Continuing Calibration Verification (CCV)	1 in 10 or fewer samples	+/- 10%
Sample Duplicate (DU)	1 in 20 or fewer samples	<20% RPD
Matrix Spike (MS/MSD)	1 in 10 or fewer sample	90-110%

If MCP samples are digested an LCSD must also be digested within each batch of samples

9.2 All blanks must read less than the TA Buffalo quantitation limit.

9.2.1 Quantitation limits are 0.01 mg/L for aqueous samples and 1.0mg/Kg for soils and other solids.

9.2.2 Reanalyze all samples associated with an unacceptable blank unless:

9.2.2.1 Detected concentrations in the sample < PQL or

9.2.2.2 Detected concentration in the method blank < 10X amount in associated sample.

9.3 Duplicates are digested and analyzed every twenty samples or less. Matrix spikes are digested and analyzed every ten samples or less. Acceptance limits for spikes and duplicates are specified in each method (335.4/9012/4500 Cn). If a duplicate or spike falls outside the acceptance limits, the data should be reviewed to determine cause. Re-analysis may be necessary if unacceptable values are due to lab error.

9.3.1 Reanalyze the LCS /CCV.

9.3.2 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

9.3.3 If 2nd analysis is unacceptable, analyze a new ICAL.

9.4 If sample specific spikes and duplicates have not been requested by a client, you must choose one for the batch. Be sure to include all information pertinent to the analysis, such as spike levels, date of analysis, analyst's initials, weights and volumes of samples in the Cyanide logbook. Also, be sure to include any out of the ordinary observations, comments or any problems that may occur.

9.4.1 Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

9.4.1.1 LCS recovery is acceptable

9.4.1.2 Recoveries in both MS and MSD are consistent (%RSD<30)

9.4.1.3 If LCS is unacceptable – re-analysis is required.

9.4.1.4 If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case.

Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

9.5 A non-conformance memo (NCM) must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

9.5.1 Holding times exceeded

9.5.2 Insufficient sample volume for reanalysis

9.5.3 In the event of unknown positives or sample matrices which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action.

9.6 Instrument QC

9.6.1 Initial Calibration Verification (ICV)

9.6.1.1 A calibration curve is made by preparing a series of standards and a blank, using suitable volumes of the Intermediate Calibration Standard

9.6.1.2 The Intermediate Calibration Standard: 2.5ml stock 1000ppm STD into 50 ml of deionized water. The 50ppm intermediate standard for instrument analysis can be used.

9.6.1.3 The calibration curve is to be digested if using a complex cyanide standard.

If a free cyanide standard is used to prepare the calibration curve then digestion is not required.

9.6.1.4 Initial Calibration Verification (ICV): Is run from the digested 0.25ppm LCS in the first batch of digested samples. This insures accuracy when curving with free cyanide intermediate and testing that curve with complex cyanide intermediate.

9.6.1.5 Calibration Curve is prepared using the following:

Curve Concentration	Intermediate STD into 50ml of 0.25N NaOH
0 mg/L	50ml of 0.25N NaOH
0.01 mg/L	0.01 ml Intermediate STD
0.05 mg/L	0.05 ml Intermediate STD
0.10 mg/L	0.1 ml Intermediate STD
0.20 mg/L	0.2 ml Intermediate STD
0.30 mg/L	0.3 ml Intermediate STD
0.40 mg/L	0.4 ml Intermediate STD
0.50 mg/L	0.5 ml Intermediate STD

A calibration curve containing these nine points is analyzed monthly, or when new reagents are made, whichever comes first.

9.6.1.6 If RCP/MCP samples are to be analyzed a calibration curve needs to be analyzed prior to analysis of samples.

9.6.2 Continuing Calibration Verification (CCV)

9.6.2.1 Continuing Calibration Verification (CCV) is made by adding 0.25mL of the 50ppm intermediate to 50mL of 0.25N NaOH for a final concentration of 0.25ppm.

9.6.2.2 A CCB and CCV must be run every ten samples on the instrument

9.6.3 Calibration Acceptance Summary

Step	Standards	Type	Control Limit	Frequency
Initial Cal	0.0-0.5mg/L	Linear	R ² -value >0.995	Once every month at a minimum
ICV/CCV/LCS	0.4mg/L 0.25mg/L(ICV) 0.1mg/L	Linear	90-110%	Once after a new curve
ICB/CCB/MB	<.01	Linear	<RL	Every 10 samples or fewer
Low Level CLP Std	.01mg/L		70-130%	Once, immediately after calibration has been run

9.6.3.1 ICAL: Acceptance criteria for the calibration curve is a correlation coefficient (R-value) ≥ 0.995 . If the R-value is less than 0.995, the calibration standards must be remade and a new curve analyzed.

9.6.3.2 The calibration curve must be analyzed at a minimum of once every month, unless CLP samples are being analyzed. If a batch contains CLP samples, a calibration curve must be run, along with a .01mg/L low level standard, before samples can be analyzed.

9.6.3.3 ICV/CCV: Obtained values must be within $\pm 10\%$ of true value.

9.6.3.4 Low-Level CLP Standard (.01mg/L): To be run immediately after calibration curve has been analyzed. Standard is made by adding 10uL of intermediate standard to 50mL of .25N NaOH. If standard is not within 70-130% acceptance limits, calibration must be run again, with low-level standard run immediately after.

10.0 Procedure

10.1 Sample Pretreatment

10.1.1 Oxidizing agents, such as chlorine, decompose most cyanides. Test by placing a drop of sample on a strip of potassium iodide (KI) starch paper previously moistened with acetate buffer solution, pH 4. A bluish color indicates the need for treatment. Add L-ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper, adding an additional 0.006g of ascorbic acid for each 100 mL of sample volume.

10.1.2 Oxidized products of sulfide convert CN^- to SCN^- rapidly, especially at high pH. Test for S_2^- by placing a drop of sample on lead acetate paper previously moistened with acetic acid buffer solution, at a pH of 4. Darkening of the paper indicates presence of S_2^- . Add lead acetate, or if the S_2^- concentration is too high, add powdered lead carbonate $[\text{Pb}(\text{CO}_3)_2]$ to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate paper. Filter sample before raising pH for stabilization. When particulate, metal cyanide complexes are suspected, filter solution before removing S_2^- . Reconstitute sample by returning filtered particulates to the sample bottle after the S_2^- removal. Homogenize particulates before analysis.

10.1.3 Aldehydes convert cyanide to cyanohydrin. Longer contact times between cyanide and the aldehyde and the higher ratios of aldehyde to cyanide both result in increasing losses of cyanide that are not reversible during analysis. If the presence of aldehydes is suspected, stabilize with NaOH at time of collection and add 2ml 3.5% ethylene diamine solution per 100 ml of sample.

10.2 Cyanide amenable to chlorination

10.2.1 When determining cyanide amenable to chlorination part of the sample is first chlorinated to decompose the cyanides. Both the chlorinated and untreated samples are then subjected to distillation. The difference between the CN^- concentrations found in the two samples is expressed as cyanides amenable to chlorination.

10.2.2 To prepare amenable portion of sample, pour 50ml of sample into a 250ml Erlenmeyer flask completely wrapped in aluminum foil. To this add $\text{Ca}(\text{OCl})_2$ solution dropwise while agitating and maintaining a pH of 11-12 (adding NaOH if necessary.) Test for proper chlorination by placing a drop of solution on a strip of KI-starch paper. Cover flasks with aluminum wrapped watch glass and maintain residual chlorine for 1 hour while agitating. After 1 hour remove any residual chlorine by dropwise addition of NaAsO_2 solution or H_2O_2 , followed by 4 drops of $\text{Na}_2\text{S}_2\text{O}_3$ solution. Test with KI-starch paper after each addition, until there is no color change. Distill as outlined in section 10.8.1

CAUTION!: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, it is necessary that this reaction be performed in a hood.

10.3 Standards, blanks, and spikes:

10.3.1 Add 0.4 mL of the 50 mg/L std, dilute to 50 mL with deionized water for a final concentration of 0.4ppm. Add 0.25mL of 50ppm Std to 50mL of DI for 0.25ppm. Add 0.1mL of 50ppm std to 50mL DI for 0.1ppm std. Reference 7.19 for how LCSs should be digested.

10.3.1.1 If soil samples are being distilled, a purchased soil ERA QC Check STD must also be added to the run. To do this, add approximately 0.5g of a solid ERA and dilute to 50 mL with deionized water.

10.3.2 Add 50 mLs of deionized water to one test tube. This is the preparation blank.

10.3.2.1 If soil samples are being digested prepare a second blank by adding 50mL of deionized water and 0.5g Ottawa sand to a test tube. This is the soil matrix preparation blank.

10.3.3 Add 50 mLs of each sample to the appropriate test tube.

10.3.3.1 For aqueous samples: Pour 50mLs sample from bottle into class A volumetric cylinder, stopping when meniscus reads 50mL.

10.3.3.2 For soil samples: Weigh 0.5 + 0.1 grams of sample. Record weight in digestion log. Transfer soil sample and bring to a final volume of 50 mL with deionized water in distillation tube.

- 10.3.4** For sample spikes, add 0.1mL of the 50mg/L intermediate standard and dilute to 50 mL with your sample (This is a 0.1mg/L spike for aqueous samples).
- 10.4** Turn on the heating blocks: allow blocks to heat up to $125^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Blocks need to be verified that they are up to temp before every digestion and recorded in the spots provided in the logbook.
- 10.5** Place one reflux impinger in each sample tube.
- 10.5.1** Place the tube containing the sample into the heat block holes, making sure the gas outlet is facing the rear of the heating unit.
- 10.6** Add 50 mLs of 0.25N NaOH into a clean tube, place one absorber impinger in each of the tubes.
- 10.6.1** Place the tube containing 0.25N NaOH in the front of the block. Connect the absorber impinger gas inlet tube to the reflux impinger gas outlet tube. Connect the impinger vacuum tube to the valve tube.
- 10.7** Place one cold finger condenser into each reflux impinger, making sure that the water ports are facing the rear of the block.
- 10.7.1** Turn on the cooling water and adjust pressure so that it reads ~35.
- 10.8** Turn on the vacuum unit and adjust each valve to give a flow of ~3 bubbles per second in each reflux tube.
- 10.9** Total and Amenable Cyanide samples:
- 10.9.1** Through the air inlet of the reflux impinger add 2.0mL of Sulfamic acid and rinse with DI. (wait 3 minutes) SLOWLY, add 5.0 mL of 1:1 sulfuric acid and rinse with DI. (wait 3 minutes) Then add 2.0 mLs of magnesium chloride solution. Rinse the air inlet with DIH₂O to ensure mixing.
- 10.10** Weak Acid Dissociable Cyanide samples:
- 10.10.1** Do not add sulfamic acid, sulfuric acid or magnesium chloride solution. Through the air inlet of the reflux impinger add 2.0 mL of acetate buffer and rinse (wait 3 minutes), and 2.0mL of zinc acetate solution. Add 2-3 drops of methyl red indicator. If the solution does not turn pink add glacial acetic acid dropwise until pink color persists. Rinse the air inlet tube with DIH₂O between reagent additions to ensure mixing.
- 10.10.2** Physiological Available Cyanide samples:
- 10.10.3** Samples are poured into the tubes with no pre-treatment (pH must be >12)

- 10.10.4** A LCS-N must be analyzed to ensure that the metal cyanide complexes are not being digested during this process. A LCS-N is 5mL of Prussian Blue intermediate standard taken up to a final volume of 50mL in a sample tube. The recovery range for the LCS-N is 0-10%. If 10% is exceeded then the batch must be reanalyzed.
- 10.10.5** The temperature of digestion is at 78°C and the digestion time is one hour from the start of air purging. Check the air flow on the bubblers of the distillation apparatus every 15 minutes to ensure that minimum flow is achieved throughout the entire process.
- 10.10.6** Do not add sulfuric acid or indicator. Through the air inlet of the reflux impinger, add 2mL of sulfamic acid (Wait 3 minutes), add 5mL of PAC buffer, and then finally add 2mL of magnesium Chloride. Rinse Inlet with DI H₂O after each added reagent.
- 10.10.7** After digestion, the sample must be checked for pH and recorded. The digested sample is to be checked for sulfide. Comment on the sample if sulfide is found.
- 10.11** Allow samples to reflux for 90 minutes. Discontinue heating and allow samples to cool with air pump on for 15 minutes.
- 10.12** Remove the absorber impinger from the tube and blow out excess liquid with bulb. Repeat for all samples.
- 10.13** Automated Analysis System Start-up Procedure
- 10.12.1** Prepare reagent and standards as described in section 10.0.
- 10.13.1** Set up manifold as shown in Attachment 17.1.
- 10.13.2** Input data system parameters as in Attachment 17.2.
- 10.13.3** Place samples and/or standards in autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.
- 10.13.4** Calibrate the instrument by injecting the standards.
- 10.14** System Notes
- 10.14.1** Allow 15 minutes for heating unit to warm up to 60°C.
- 10.14.2** If sample concentrations are greater than 0.5 mg/L, the digested sample should be diluted with 0.25N NaOH diluent. Do not dilute digested samples or standards with Di Water.

10.14.3 For analyzing digested samples, prepare a standard curve by plotting the peak area of standards against known concentration values. Compute concentrations by comparing sample peak area with standard curve.

11.0 Calculations / Data Reduction

11.1 Sample results are calculated from the calibration curve by using linear regression.

11.2 In the case of any dilution, the results do not have the dilution factor automatically calculated.

11.3 For liquid samples, the result is expressed as mg/L.

11.4 For solid samples, the result is expressed as mg/Kg on a dry basis.

11.5 To convert the mg/l result obtained from the calibration curve to mg/Kg use the following equation:

$$\text{mg/Kg (wet)} = [\text{mg/L} \times \text{final vol. of leached sample}] / \text{grams sample used}$$

$$\text{mg/Kg (dry)} = \text{mg/g (wet)} / \text{decimal dry weight}$$

11.6 Percent Recovery for Analyses Involving Spikes:

$$\% \text{ Recovery} = \left[\frac{(\text{SSR} - \text{SR})}{\text{SA}} \right] \times 100$$

where:

SSR = spiked sample result
SR = sample result
SA = spike added

11.7 Relative Percent Difference (RPD):

$$\text{RPD} = \frac{\left| \frac{x_1 - x_2}{\frac{x_1 + x_2}{2}} \right|}{2} \times 100$$

where:

x_1 = analytical % recovery
 x_2 = replicate % recovery

11.8 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

- a = area counts for analyte to be measured
- m = slope
- x = concentration
- b = intercept

11.9 Percent Recovery for LCS:

$$\% \text{ Recovery (LCS)} = 100 \left(\frac{E}{C} \right)$$

where:

- E = obtained (experimental) value
- C = true value

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See TestAmerica SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current TestAmerica Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

12.2 Demonstration of Capabilities

- 12.2.1 A one-time initial demonstration of performance for each individual method for both soil and water matrices must be generated.
- 12.2.2 This requires quadruplicate analysis of a mid-level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.
- 12.2.3 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 12.2.4 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

- 12.2.5** Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

- 12.3.1** The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 12.3.2** The following analyst validation information is maintained for this method in the laboratory QA files.
- 12.3.3** The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- 12.3.4** The analyst must read and understand this SOP.
- 12.3.5** The analyst must read and understand the Method used as reference for this SOP.
- 12.3.6** The analyst must complete a DOC or successfully analyze PT samples annually.
- 12.3.7** The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

- 13.1** Waste Streams Produced by the Method from the instrument contains pyridine, which has a pH >7 and is disposed of in the "D Pyridine" waste container.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.0 References / Cross-References

- 15.1** USEPA Methods for the Determination of Inorganic Substances in Environmental Samples, Method 335.4, EPA/600/R-93/100, Revised August 1993
- 15.2** Method 9010B, "Total and Amenable Cyanide: Distillation," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Update III, December 1996.

- 15.3** Method 9012A, "Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Third Edition, Update III, September 1996.
- 15.4** Method 9012B, "Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Third Edition, Update III, August 2002.
- 15.5** Physiologically Available Cyanide (PAC) Method, Massachusetts Department of Environmental Protection, January 1996 Protocol.
- 15.6** Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012, Method 4500-CN E and 4500-CN G.

16.0 Method Modifications:

Item	Method	Modification
1	Lachat 10-204-00-1	Solutions are not degassed. There is no interference from gas in the solutions or reagents

17.0 Attachments

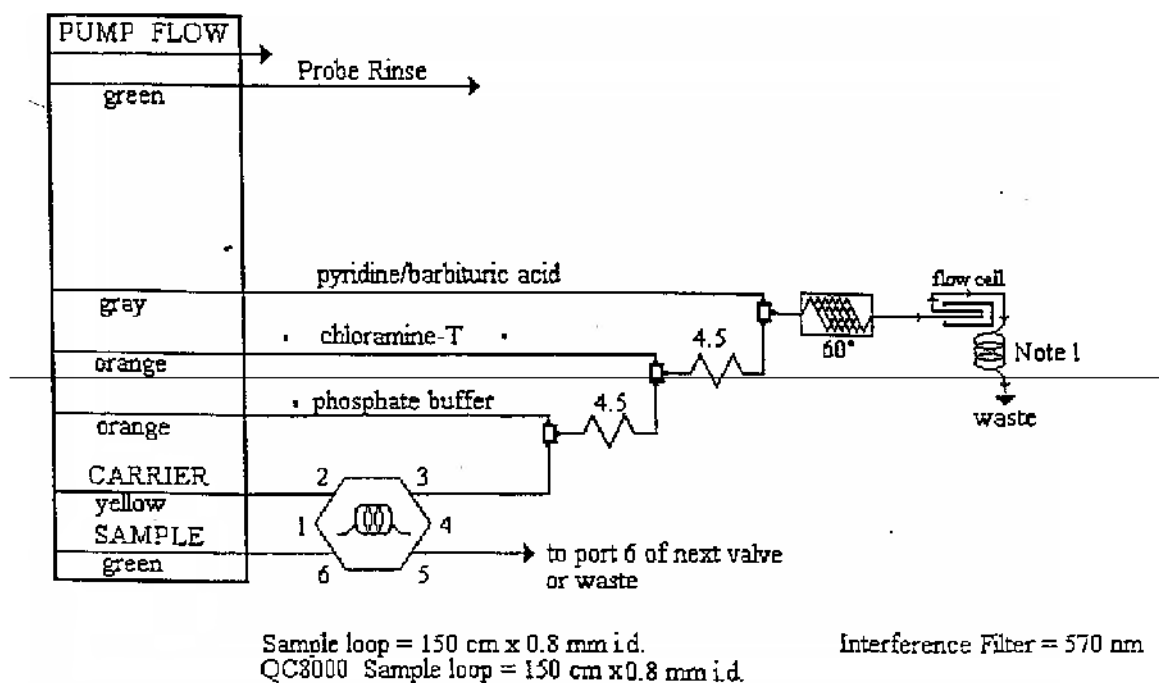
- 17.1:** Cyanide Manifold Diagram
- 17.2:** Data System Parameters for QC8000
- 17.3:** Analytical Sequence
- 17.4:** Analytical Batch
- 17.5:** Digestion Batch
- 17.6:** Wet Chemistry Batch Summary

18.0 Revision History

- Revision 9, dated 13 August 2015
 - o Wording updated for sections 7.14 and 7.15
 - o Sections 7.19, 7.19.1, 9.6.3, 10.3.1 updated to include all LCS values
 - o Section 7.19.2 removed RCP from LCSD requirement
 - o Added section 7.8 to include soil LCS ERA reagent
 - o Updated section 9.1 to include DU/MS/soil LCS requirement
 - o Section 9.5 updated to say NCM
 - o Section 9.6.1.4 added to include ICV
 - o Section 10.3.3.1 added the use of a Class A Volumetric flask.
 - o Section 10.3.4 removed mention of SRM
 - o Section 10.8.1 added rinses
 - o Added section 10.4 for block temperatures
 - o Sections 10.10.3-10.10.7 made into 10.10.2.1-10.10.2.5
 - o Section 10.11 updated reflux times
 - o Updated attachments 17.3, 17.4, and 17.5
 - o Changed Department Manager and Laboratory Director, signatures changed.

- Revision 8, dated 12 June 2014
 - o Section 7.11 – Chloramine T acid to be made daily.
 - o Section 7.12 -- Pyridine barbituric acid to be made weekly.
 - o Section 10.8.1/10.9.1 – Rinsing of the air inlet and timing of additions changed.
 - o Section 10.11 – Reflux time changed to 90 minutes.
- Revision 7, dated 19 July 2013
 - o Updated Department and Quality manager signatures.
 - o Section 4.5 added to reflect additional interferences.
 - o Matrix spike required every ten samples instead of twenty samples
 - o PAC added to SOP: Sections 7.19, 7.20, 7.21, 10.10, 15.5
- Revision 6, dated 18 September 2012
 - o Added method 9012B to Title.
 - o In Section 15.0 added Method 9012B reference.
- Revision 5, dated 24 July 2012
 - o Updated Section 7.0, Reagents and Standards
 - o Updated Sections 9.1 and 9.6.1.5 for RCP/MCP requirements
 - o Updated Sections 10.3.1, 10.3.2 for soil procedures
 - o Updated Attachment 17.3
 - o Updated Attachment 17.6
- Revision 4, dated 28 June 2011
 - o Removed ERA references for water, no longer used in digestion for water samples
 - o Updated CCV/CCB frequency and run information
 - o Updated Attachments 17.3, 17.4, 17.5
 - o Changed Quality Manager, signature added.
- Revision 3, dated 28 Jan 2010
 - o Updated Attachments 17.4, 17.5
- Revision 2, dated 31 July 2009
 - o Updated shelf life of Chloramine-T reagent to 1 week
 - o Updated Attachments 17.4, 17.5
- Revision 1, dated 18 July 2008
 - o Added CLP information. Replaces Total Cyanide CLP-Cn SOP
 - o Updated department manager and QA manager signatures
 - o Updated Section 7.0 to reflect refrigeration of reagents
 - o Updated Attachment 17.6

Attachment 17.1 Cyanide Manifold Diagram



Carrier is 0.25 N sodium hydroxide solution. All manifold tubing is 0.8mm (0.030 in) i.d. This is 5.2 uL/cm. 4.5 is 70 cm tubing on a 4.5 cm coil support.

APPARATUS: An injection valve, a 10mm path length flow cell, and a colorimetric detector module



are required. The

shows 650 cm tubing wrapped around the heater block at the specified

Note 1: 2 meter back pressure loop, 0.52 mm i.d.

Attachment 17.2 Data System Parameters for QC 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample Throughput: 80 samples/h, 45 s/sample
Pump speed: 35
Cycle period: 45

Analyte Data:

Concentration Units: mg CN-/L
Peak Base Width: 39s
% Width Tolerance: 100
Threshold: 25000
Inject to Peak Start: 24 s
Chemistry: Direct

Calibration Data:

Levels	1	2	3	4	5	6	7	8	9
Concentration mg/L	0.50	0.40	0.30	0.20	0.10	0.05	0.01	0.00	0.00

Calibration fit Type: 1ST Order Polynomial
Calibration Rep. Handling: Replace
Weighting method: No
Concentration Scaling: None
Force through Zero: No

Sampler Timing:

Min. Probe in wash Period: 14 s
Probe in Sample Period: 20 s

Valve Timing:

Load Time: 0.0 s
Load Period: 20 S
Inject Period: 25 s

Attachment 17.3 Analytical Run Sequence

Cyanide Non CLP sequence

CCV	
CCB	Second digestion
MB	MB
LCS 0.4	LCS 0.25
LCS 0.25	LCS 0.1
LCS 0.1	Sample
Sample	Ect.
Sample	
Sample	
Sample	
Sample	
Sample spike (MS)	
CCV	
CCB	
Sample	
Sample	
Sample	
Sample	
Sample	
Sample	
Sample	
Sample	
Sample duplicate (MD)	
Sample spike (MS)	
CCV	
CCB	

Cyanide MCP sequence

CCV
CCB
MB
LCS 0.25
LCSD 0.25
LCS 0.1
Sample
Sample
Sample
Sample
Sample
Sample spike (MS)
CCV
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate (DU)
Sample spike (MS)
CCV
CCB

Attachment 17.3
Analytical Run Sequence continued

Cyanide PAC sequence

CCV
CCB
MB
LCS
LCSN
Sample
Sample
Sample
Sample
Sample
Sample
Sample Spike (MS)
CCV
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate(DU)
Sample Spike (MS)

Attachment 17.4 Analytical Batch Run

Author: butlachat2

Date: 8/10/2015

Original Run Filename: OM_8-10-2015_02-57-31PM.OMN Created: 8/10/2015 2:57:31 PM
Original Run Author's Signature: [butlachat2]
Current Run Filename: OM_8-10-2015_02-57-31PM.OMN Last Modified: 8/10/2015 3:34:17 PM
Current Run Author's Signature: [butlachat2]
Description: 10-204-00-1-A

Sample	Rep.	Cup No.	Channel 1 Cyanide			Detection Time
			Conc. (mg/L)	Area (V.s)	Height (V)	
CCV	1	S9	0.271	25.5	0.764	8/10/2015@2:58:14 PM
Known Conc:			1.50			
Calibration:			Table/Plg: 1			
CCB	1	S10	4.07e-3	-0.122	5.74e-3	8/10/2015@2:59:40 PM
Known Conc:			0.00			
MB 480-257892/1-A	1	1	5.01e-3	-0.0310	-1.82e-3	8/10/2015@3:01:07 PM
LCS 480-257892/2-A	1	2	0.397	37.6	1.05	8/10/2015@3:02:34 PM
LCS 480-257892/3-A	1	3	0.255	24.0	0.670	8/10/2015@3:04:01 PM
LCS 480-257892/4-A	1	4	0.0957	8.87	0.237	8/10/2015@3:05:28 PM
480-85073-1-1	1	5	6.12e-3	0.0747	3.60e-3	8/10/2015@3:06:55 PM
480-85073-1-1-J MS	1	6	0.103	9.38	0.258	8/10/2015@3:08:21 PM
480-85073-1-1-K MSD	1	7	0.108	9.84	0.275	8/10/2015@3:09:48 PM
440-116907-B-2-C	1	8	0.0909	8.21	0.231	8/10/2015@3:11:14 PM
480-85185-N-3-A	1	9	0.0136	0.793	0.0230	8/10/2015@3:12:40 PM
480-85053-AA-1-A	1	10	0.0116	0.605	0.0157	8/10/2015@3:14:06 PM
CCV	1	S9	0.259	25.3	0.711	8/10/2015@3:15:32 PM
Known Conc:			100			
CCB	1	S10	5.28e-3	-7.57e-3	1.61e-3	8/10/2015@3:16:58 PM
Known Conc:			100			
480-85185-N-4-A	1	11	6.90e-3	0.150	5.08e-3	8/10/2015@3:18:24 PM
480-85185-N-4-B MS	1	12	0.0471	4.01	0.115	8/10/2015@3:19:50 PM
480-85185-N-5-A	1	13	9.06e-3	0.367	0.0113	8/10/2015@3:21:16 PM
480-85185-N-6-A	1	14	0.0139	0.817	0.0263	8/10/2015@3:22:41 PM
480-85185-N-7-A	1	15	6.39e-3	0.101	3.48e-3	8/10/2015@3:24:07 PM
480-85185-N-8-A	1	16	0.0196	1.08	0.0315	8/10/2015@3:25:34 PM
480-85185-N-9-A	1	17	0.0237	1.75	0.0481	8/10/2015@3:27:01 PM
480-85185-N-10-A	1	18	0.0111	0.552	0.0152	8/10/2015@3:28:28 PM
CCV	1	S9	0.266	25.2	0.707	8/10/2015@3:29:54 PM
Known Conc:			100			
CCB	1	S10	5.29e-3	-4.87e-3	1.08e-3	8/10/2015@3:31:20 PM
Known Conc:			100			

Table: 1 (Cyanide)

	Known Conc. (mg/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Residual	Det. Conc (mg/L)	Detection Date	Detection Time
1	0.500	1	47.2	1.35	0.0	-0.1	0.497	7/30/2015	10:06:56 AM
2	0.400	1	37.8	1.08	0.0	-0.4	0.399	7/30/2015	10:08:22 AM
3	0.300	1	28.1	0.805	0.0	0.1	0.298	7/30/2015	10:09:49 AM
4	0.200	1	18.4	0.528	0.0	0.6	0.198	7/30/2015	10:11:16 AM
5	0.100	1	9.38	0.268	0.0	-3.8	0.103	7/30/2015	10:12:42 AM
6	0.0500	1	4.53	0.130	0.0	-6.0	0.0525	7/30/2015	10:14:10 AM
7	0.0100	1	-0.208	-5.83e-3	0.0	145.4	3.16e-3	7/30/2015	10:15:37 AM
8	0.00	1	0.0892	2.89e-3			6.27e-3	7/30/2015	10:17:04 AM

Attachment 17.5
Digestion Logbook

TALS Batch No. TestAmerica **BUFFALO** Logbook# A15-03-08
Cyanide Distillation Logbook

Distillation Date	Analyst	Job #	Sample I.D.	Dist. Flask #	Sample Volume (ml)	Soil Weight (g)	Spike Amount	If Spiked Please ☒	Comments
8/10/15	MB	MB	Blank	1	50				
			CS	2			400	✓	
			CS	3			250	✓	
			CS	4			100	✓	
			85073	I-1	5				total
				I-1ms	6		100	✓	
				I-1msD	7		100	✓	
			440-116507	S-2	8				total Antifoam
			85185	N-3	9				Antifoam
			85053	AA-1	10				
			85185	N-4	11				
				N-4ms	12		100	✓	
				N-5	13				
				N-6	14				
				N-7	15				

Start Time 11:10 End Time 12:50 H₂S ✓ Cl₂ ✓ ERA Water Loss
Magnesium Chloride: 2882966 Cyanide Total Standard: ERA Soil Loss
Sulfuric Acid: 2891532 Cyanide Intermediate Standard 2878189 0.25N NaOH: 2895757 Cyanide Free Standard:
Acetate Buffer: Zinc Acetate: H₂O₂: 2884285
Block Temp Check:
Block# 3 Measured Temp °C 124^u Corrected Temp °C (After Correction factor has been applied) 126^u Cell# 3
Block# 2 Measured Temp °C 123^u Corrected Temp °C (After Correction factor has been applied) 123.2^u Cell# 3
Other: Antifoam 2438463 Reviewed By MB Date 8/10/15

Attachment 17.6 Wet Chemistry Batch Summary

TestAmerica [lab name]



Colorimetric Cyanide Data Review Checklist

LIMS Batch Number:	Filename:	Instrument ID (circle one):	Spec1	Spec2	
Analyst/1 st Reviewer:	Method (circle): 335.4, 9010, 9014, 4500CN-E	QC Type (circle):	Standard	QAPP Other	
Matrix (circle): Drinking Water Non-potable Water Solid Leachate		Circle One:	Total	Soluble	
Review Items	NA	Yes	No	2 nd Rev	If No, why is data reportable?
A. Calibration/Instrument Run QC					
1. Frequency: Daily (4500CN-E&G) When CCV fails (300 series and 9000 series)					
2. Number of Points: 6 standards & blank (9000 series) 1 st order: 3 standards & blank (4500CN-E&G, 300 series) 2 nd order: 6 standards & blank (4500CN-E&G, 300 series)					
3. Linearity and Intercept: $r \geq 0.995$ a. $ x\text{-Intercept} < RL$ or b. $< \frac{1}{2} RL$ (special projects)					
4. ICV, second source: run before samples 90-110% recovery (300 series) 85-115% recovery (9000 series and 4500CN-x)					
5. CCV: 10% frequency & closing 90-110% recovery					If no, list details:
6. Distilled check standard, high and low concentration: Required if ICAL not distilled (300 series, 9000 series) 90-110% recovery					If no, list details:
7. IC8/CCB: run before samples, 10% frequency, & closing Result < RL Result < $\frac{1}{2} RL$ (DoD or special project)					If no, list details:
B. Client Sample and QC Sample Results					
8. Samples with results > linear range diluted and reanalyzed?					Comments:
9. On-instrument response of diluted sample is >10X MB on-instrument response					Comments:
C. Preparation/Matrix QC					
10. Method Blank: one per preparation batch Result < RL Result < $\frac{1}{2} RL$ (DoD or special project) If no, list blank ID & explain:					☐ No analyte > RL in associated samples ☐ Sample results >10x blank ☐ Insufficient sample for reanalysis
11. LCS: one per preparation batch 90-110% recovery (300 series) 85-115% (9000 series) Lab limits (4500CN-x) If no, list LCS ID & explain:					☐ Insufficient sample for reanalysis ☐ LCS %R > QC limits & samples < RL
12. MS/MSD or MS/Dup frequency: A pair per 10 samples (300 series, 9000 series) A pair per batch (4500CN-x) If no, list QC ID & explain:					☐ Insufficient sample
13. MS/MSD recovery & RPD: 80-120% recovery; 20% RPD If no, list MS or MSD ID & explain:					☐ LCS acceptable -- matrix effects ☐ Native analyte > 4x spike level ☐ Matrix effect and native analyte > 4x spike

TestAmerica [lab name]

Review Items	Yes	No	2 nd Rev	If No, why is data reportable?
D. Raw Data & TALS Data Entry				
14. Raw Data				
a. Unused data is clearly identified				
b. All crossed out data is initialed and dated				
c. Out of control QC is clearly identified				
d. Any data that has a qualifier tick is commented on with appropriate action taken				
e. The first page of the run includes the filename, instrument, and analyst initials/signature				
15. Run Log				
a. Unused data is clearly identified				
b. All crossed out data is initialed and dated				
c. Analyst initials/signature provided				
16. TALS Samples Tab				
a. LIMS Sample IDs / Containers are correct				
b. Method and matrix are correct				
c. Date and time match raw data				
d. Dilutions are correct				
e. Correct suffix designated (where applicable)				
17. TALS Worksheet Tab is complete and correct				
18. TALS Reagent Tab is complete and correct				
19. TALS QC Links Tab is correct				
20. TALS Sample Results Tab				
a. All unused data are marked Rejected or Accepted				
b. All reported analytes are marked Primary or Secondary				
21. TALS Batch Information Screen documentation is complete				
22. TALS Status set to appropriate review level				
E. Final Report and NCMs (2nd level review only)				
23. Were all job/project requirements met?				
24. Results for samples and QC correct on final report?				
25. Are all necessary scanned documents in TALS?				
26. NCMs reviewed for applicability, correct references to batches, grammar/typographical errors?				

2nd Reviewer: _____ Review Date: _____

Comments: _____

**Title: HEXAVALENT CHROMIUM
7196A, SM3500-Cr B & SM3500-Cr D**

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):



Jennifer Pierce
Department Manager

6/22/15

Date



Kenneth Kasperek
Laboratory Director

6/22/15

Date



Brad Prinzi
Quality Assurance Manager

6/22/15

Date

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1.0 Scope and Application

The hexavalent chromium is determined colorimetrically by a reaction with diphenylcarbohydrazide in an acid solution. A red violet color of unknown composition is produced.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is taken from the EPA approved HACH 1,5 Diphenylcarbohydrazide method, SW 864 3rd Edition Method 7196A, Standard methods 3500 Cr-B and 3500Cr-D.

This method is applicable to an aqueous matrix.

The reporting limit has been determined to be 0.01 mg/l for aqueous samples.

2.0 Summary of Method

Hexavalent chromium is determined by the 1,5 diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains a buffer combined with 1,5 diphenylcarbohydrazide, which reacts to give a purple color when hexavalent chromium is present.

3.0 Definitions

Standard definitions are found in the laboratory Quality Manual.

4.0 Interferences

The reaction with diphenylcarbohydrazide is nearly specific for chromium.

Hexavalent molybdenum and mercury salts will react to form color with the reagent but the intensities are much lower than that for chromium at the specific pH.

Vanadium interferes strongly but concentrations up to 10 times that of chromium will not cause problems.

Since this method measures suspension, color or suspended salts may cause a positive interference. Correct by running sample blanks from which the 1,5 diphenylcarbohydrazide has been omitted and subtract the absorbency from the treated sample.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially

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

5.1 Specific Safety Concerns or Requirements

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

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material ¹	Hazards	Exposure Limit ²	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
¹ – Always add acid to water to prevent violent reactions.			
² – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Genesys 20 Spectrophotometer or equivalent for use at 540nm.

6.2 Supplies

- Volumetric class A flask, 50ml and 25ml.

- pH indicator paper, 0 to 2.5 ranges.
- Graduated cylinder, 25mls.
- Test tubes

7.0 Reagents and Standards

7.1 Water, deionized.

7.2 For Calibration Curve: Ultra Scientific Single element Standard Chromium $K_2Cr_2O_7$ as Cr^{+6} in water $1000 \pm 3\mu g/ml$.

7.2.1 10ppm Intermediate Standard: Add 250ul of 1000ppm $K_2Cr_2O_7$ as Cr^{+6} into a 25ml volumetric flask and dilute to 25mls with deionized water.

7.3 Second Source Standard: ERA Single Element Chromium $K_2Cr_2O_7$ standard must also be purchased from a second vendor to provide a second source. If a second vendor is not available a second lot number from the initial vendor must be used.

7.3.1 10ppm Intermediate Standard: Add 250ul of 1000ppm $K_2Cr_2O_7$ as Cr^{+6} into a 25ml volumetric flask and dilute to 25mls with deionized water.

7.4 ChromaVer 3 Chromium Reagent Powder Pillow (manufactured by HACH).

7.5 Sulfuric acid, ACS.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Plastic	100mLs	Unpreserved	24 hours	SW-846 3 rd Edition

8.1 Collect samples in a cleaned glass or plastic container and refrigerate at $4\pm 2^\circ C$ if analysis is to be delayed.

9.0 Quality Control

9.1 Sample QC

9.1.1 An acceptable calibration verification sample and calibration blank must bracket each group of sample analyses. All quality control data should be maintained and available for easy reference or inspection.

9.1.2 Calibration Curve: A calibration curve, prepared using Ultra Scientific Standard (section 7.2 and 7.2.1) must be run at a minimum of once every three months, or earlier if needed, and the curve correlation must be 0.995 or better.

9.1.3 Initial Calibration Verification (ICV): (0.2mg/L) Prepare an initial calibration verification standard by using 0.500ml of the ERA $K_2Cr_2O_7$ Intermediate Standard

(see section 7.3 and 7.3.1) from a standard purchased from a second source vendor, added to 25mLs of reagent water. Analyze one ICV once after a new calibration curve has been analyzed. The ICV must be within 85 - 115% or the standards remade and the calibration curve reanalyzed.

9.1.4 Continuing Calibration Verification (CCV) Waters: (0.05mg/L) Prepare a calibration standard using 0.125ml of the diluted ERA $K_2Cr_2O_7$ Intermediate Standard (see section 7.3.1) added to 25mLs of reagent water. Analyze a CCV once at the beginning and end of the analytical batch and after every ten samples within the analytical batch. The CCV must be within 85-115%. If the CCV falls outside of this range, the standards must be remade and/or the calibration curve reanalyzed.

9.1.5 Laboratory Control Sample (LCS) Waters: (0.05mg/L) Prepare a calibration standard using 0.125ml of the diluted ERA $K_2Cr_2O_7$ Intermediate Standard (see section 7.3.1) added to 25mLs of reagent water. Analyze an LCS once at the beginning and end of the analytical batch and after every ten samples within the analytical batch. The LCS must be within 85-115%. If the LCS falls outside of this range, the standards must be remade and/or the calibration curve reanalyzed.

9.1.6 Blanks: To determine freedoms from contamination analyze one Continuing Calibration Blank (CCB) at the beginning and end of the analytical batch and after every 10 samples within the analytical batch. Analyze one Method Blank (MB) at the beginning of each analytical batch and after every 20 samples. Use 25mLs of laboratory reagent water carried through the entire analytical procedure. The calibration blank must exhibit values less than the reporting limit (0.01 mg/L). If the blanks are not less than the quantitation limit the analytical results for each sample must be evaluated.

9.1.7 **Matrix Duplicate:**

9.1.6.1 Aqueous samples must have a matrix duplicate sample analyzed every 10 samples. The Relative Percent difference (RPD) must be $\leq 15\%$.

9.1.8 **Matrix Spike:**

9.1.8.1 Aqueous samples must have a matrix spike analyzed at a frequency of one per ten samples at a 0.05ppm. Prepare a matrix spike by using 0.125mL of the diluted ERA $K_2Cr_2O_7$ Intermediate Standard (see section 7.3.1) added to 25mLs of sample. Use the same standard as the ICV. The acceptance range for matrix spike recoveries is 85-115%. If the spike is outside of the recovery limits, but the associated quality control standards are within criteria, the analytical results for the spike must be evaluated.

9.1.8.1.1 If the MS or MS/MSD indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference is still present after dilution, comment in TALS on the matrix interference.

9.1.8.2 For every sample matrix analyzed, a MS must be analyzed to ensure that neither a reducing condition nor chemical interference is affecting color development.

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

Waters:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	85-115%
Matrix Spike (MS) ¹	1 in 10 or fewer samples	85-115%
Matrix Duplicate (MD) ¹	1 in 10 or fewer samples	85-115%
Continuing Calibration Verification (CCV)	1 in 10 or fewer samples	85-115%
Continuing Calibration Blank (CCB)	1 in 10 or fewer samples	< Rpt. Limit

¹ The sample selection for MD/MS is random unless specifically requested by a client.

9.2 Instrument QC

9.2.1 Calibration Acceptance Summary

Step	Standards	Type	Control Limit	Frequency
<i>Method # 7196A</i>				
Initial Cal	0.0PPM-0.4PPM	Linear	85-115%	Every 3 months, at a minimum
ICV	0.2 mg/l	Linear	85-115%	Immediately after calibration
ICB	< .01	Linear	< RL	Immediately after calibration

10.0 Procedure

10.1 Analysis

10.1.2 Fill a sample cup with 25mLs of sample.

10.1.3 Add the contents of a ChromaVer 3 reagent Powder Pillow to the cup containing the sample. Swirl to mix. A purple color will form if hexavalent chromium is present. At high chromium levels precipitate will form; dilute sample according to sample dilution techniques if needed.

10.1.4 After a reaction time of five minutes check the pH of the sample using pH strips. Record the pH check in the appropriate column on the spreadsheet. The pH must be 1.5-2.5. If the pH is outside of this range it must be adjusted with H₂SO₄. Pour off another aliquot of sample, adjust the pH, take a blank absorbance and re-analyze.

- 10.1.5** Fill the cuvette with prepared sample and insert into spec.
- 10.1.6** Set the instrument wavelength for 540nm. Let the instrument equilibrate and wait for your absorbance to stop fluctuating.
- 10.1.7** When the absorbance 'levels off' record on the spreadsheet.
- 10.1.8** If a result above the upper point of the calibration curve is recorded, samples must be centrifuged and reanalyzed at an appropriate dilution.
- 10.1.9** A blank correction must be done for all colored samples. To blank correct the samples fill a sample cell with 25mLs of sample (the blank correction). This sample is not spiked nor do you add the ChromaVer 3 reagent powder pillow. Place the blank correction sample into the cell holder and record the absorbance.

****All hexavalent chromium spreadsheets can be found in the Wet Chemistry Spreadsheets folder located in the L:/ drive****

10.2 Calibration

10.2.1 Hexavalent Chromium Calibration Curve

Concentration	Curve Standard
0.000ppm	0.000mls Intermediate Standard diluted to 25mls with DI-water.
0.010ppm	0.025mls Intermediate Standard diluted to 25mls with DI-water.
0.020ppm	0.050mls Intermediate Standard diluted to 25mls with DI-water.
0.050ppm	0.125mls Intermediate Standard diluted to 25mls with DI-water.
0.100ppm	0.250mls Intermediate Standard diluted to 25mls with DI-water.
0.200ppm	0.500mls Intermediate Standard diluted to 25mls with DI-water.
0.300ppm	0.750mls Intermediate Standard diluted to 25mls with DI-water.
0.400ppm	1.000mls Intermediate Standard diluted to 25mls with DI-water.

10.2.2 A second source of $K_2Cr_2O_7$ must be used for the curve, and it can not be the same vendor as the standard used for the ICV/CCV's and the spikes. If a second vendor is not available a second lot number from the initial vendor must be used.

10.2.3 A calibration curve must be run every 3 months at a minimum.

10.2.4 The curve correlation must be 0.995 or better.

10.3 MCP/RCP Requirements

10.3.1 Calibration is to be performed prior to analysis of samples

10.3.2 ICV/CCV/LCS will be analyzed at 0.2mg/L (mid-point of the calibration curve.)

10.3.2.1 0.500mL of Intermediate standard diluted to 25mL with DI H2O

10.3.3 LCS will be analyzed in duplicate, unless an MCP sample is analyzed in duplicate (MD)

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

- 11.3** To obtain Cr⁺⁶ concentrations in mg/L enter the absorbencies of each sample into the spreadsheet, which contains the most current calibration curve. The spreadsheet will subtract the blank correction absorbance from the sample absorbance and calculate the final concentration.

11.3.1 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

Where:

- x = concentration
- a = area counts for analyte to be measured
- b = intercept
- m = slope

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

A valid method detection limit for each matrix must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See TestAmerica SOP, "Method Detection Limit Studies," current revision, for further guidance. Current TestAmerica Buffalo MDLs are maintained by the QA department and are easily viewed in the laboratory LIMS system.

12.2 Demonstration of Capabilities

A one-time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

- 12.2.1** This requires quadruplicate analysis of a mid-level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.
- 12.2.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 12.2.3** Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).
- 12.2.4** Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

- 12.3.1** The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 12.3.2** The following analyst validation information is maintained for this method in the laboratory QA files.
- 12.3.3** The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- 12.3.4** The analyst must read and understand this SOP.
- 12.3.5** The analyst must read and understand the Method used as reference for this SOP.
- 12.3.6** The analyst must complete a DOC or successfully analyze PT samples annually.

13.0 Pollution Control

- 13.1** It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated.

14.0 Waste Management

- 14.1** All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 14.2** The following waste streams are produced when this method is carried out.
 - 14.2.1** Acidic waste generated by the analysis. All waste generated from this analysis is disposed of into "AN" waste.

15.0 References / Cross-Reference

15.1 HACH Water Analysis Handbook, 1,5 Diphenylcarbohydrazide Method 8023.

15.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, US Environmental Protection Agency, Office of Solid Waste and Emergency Response.

15.3 Standard Methods for the Examination of Water and Wastewater, 19th Edition 3500-Cr D, American Public Health Association/American Water Works Association/Water Environment Federation, Washington, DC.

15.4 Standard Methods for the Examination of Water and Wastewater, 22nd Edition 3500-Cr B-2011.

16.0 Method Modifications:

Item	Method	Modification
1	7196A	Pre-made reagents and standards certified from independent vendor.
10.1.2	3500-Cr D	25mL sample volume is used in place of 10mLs due to the use of ChromaVer3 Chromium Reagent Powder Pillows, 25mL as per Hach Method 8024

17.0 Attachments

17.1 Analytical Run Sequence for aqueous samples.

17.2 Wet Chemistry Batch Summary.

18.0 Revision History.

- Revision 6, 06/19/2015
 - Updated section 7 to clarify two separate standards being used
 - Updated section 9 to indicate that the CCV, LCS, and MS are created from the second source and updated
 - Updated 9.1.6 to clarify frequency of MB and CCB
 - Updated QC Table in 9.1 with appropriate limits and added CCB
 - Updated Instrument QC Table in 9.2 with correct ICV concentration
- Revision 5, 01/17/2014
 - Updated sections 6.1, 9.1.8.1, 10.3.2.1 and 16.0
 - Sections 9.1.6 and 15.4 added
 - Updated methods under Title
- Revision 4, 10/7/2013
 - Sections 9.1.7.2 and 9.1.7.1.1 added.

- Updated section 9.2.1
 - Updated 10.1.4
 - Updated Department Manager and Quality Manager.
- Revision 3, 07/24/2012
 - Section 10.3 added for MCP/RCP requirements
 - Revision 2, 01/19/2012
 - Updated nomenclature for current LIMS system
 - Updated attachments
 - Changed Quality Manager, signature added
 - Revision 1, 01/25/2010
 - Removed all soil matrix methods, analysis no longer performed
 - Updated attachments; removed soil attachments
 - Revision 0, 02/18/2008
 - Updated attachments 17.2 & 17.5
 - Integration for TestAmerica Laboratory Operations

Attachment 17.1
Analytical Run Sequence

CCV
CCB
MB
LCS
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate
Sample Spike
CCV
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate
Sample Spike
CCV
CCB

Attachment 17.2 Wet Chemistry Batch Summary



Data Review Coversheet—Wet Chemistry Dept

Analysis: _____

Method: _____

Date of Analytical Run: _____

Analyst/Primary Reviewer: _____

Date of Primary Review: _____

Secondary Reviewer: _____

Date of Secondary Review: _____

Prep Batch Number					

Analytical Batch Number					

Criteria for QC	1 st Level	2 nd Level	n/a	Notes/Comments
Does the calibration meet method requirements? Low point at or below RL, minimum number of calibration points met per SOP, $r > \text{or} = 0.995$				
Was Data Imported _____ Manually Entered _____ Balance interface Used _____				
Were the ICV, CCV and LCS within acceptable limits for QC recovery?				
Were the ICB, CCB and MB all <RL?				
Was there a CCV/CCB combination run after every 10 samples or less?				
Was there an LCS run with every batch of 20 samples or less?				
Was there a DUP, MS or MSD run with every batch of 20 samples or less?				
Were all MS/MSD results within acceptable limits for QC recovery?				
Were all DUP or MSD RPDs within acceptable limits for QC recovery?				
Were the raw data points for samples within the working curve range, or if not were the samples diluted to bring them within this range?				
Are dilution factors all present and correct?				
Do all entries match raw data?				
Were there any holding time violations in this batch?				NOTE! The PM and QA Manager must be notified by email <i>immediately</i> of any holding time violations!!
Are all errors crossed out with single line, initialed and dated?				
Were any NCMs needed in the batch?				NCM #s:

WC-Data Review Coversheet
Rev. I
September 28, 2011