STUDY AREA CHARACTERIZATION WORK PLAN

Study Area Bounded by Pyrex Street, E. Pulteney Street, Post Creek and Chemung River
Corning, NY

NYSDEC Project ID 851046

June 2014

Prepared for:

Corning Incorporated
Corning, New York

Prepared by:

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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 20th day of June 2014

Weston Solutions, Inc.

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
DUSR	Data Usability Summary Report
EDD	electronic data deliverable
EM	electromagnetic
FEMA	Federal Emergency Management Agency
FOIL	Freedom of Information Law
ft amsl	feet above mean sea level
ft bgs	feet below ground surface
GPR	ground penetrating radar
GPS	global positioning system
HASP	Health and Safety Plan
IDW	investigative derived waste
in bgs	inches below ground surface
mS/m	millisiemens per meter
NWI	National Wetland Inventory
NYSDEC	New York State Department of Environmental Conservation
PAH	polycyclic aromatic hydrocarbon
PC	public-conservation zoning
PCB	polychlorinated biphenyl
PID	Photoionization Detector
PVC	polyvinyl chloride
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
R1	low-density residential zoning
RCRA	Resource Conservation and Recovery Act

LIST OF ACRONYMS (Continued)

SCO	soil cleanup objectives
SOP	standard operating procedure
SVOC	semi-volatile organic compound
TPH	total petroleum hydrocarbon
USACE	U.S. Army Corps of Engineers
USGS	U.S. Geological Survey
VOC	volatile organic compound
WESTON®	Weston Solutions, Inc.

1. INTRODUCTION

The Study Area is located in the City of Corning, New York, and is bounded by Pyrex Street on the west, E. Pulteney Street on the north, Post Creek on the east and the Chemung River on the south, as illustrated on Figure 1-1 (Study Area). During construction activities as a part of the expansion of the Corning-Painted Post East High School located in a portion of the Study Area, fill materials that the School District described as containing ash, brick, and glass waste was encountered within the excavation area.

The New York State Department of Environmental Conservation (NYSDEC) approached Corning Incorporated and presented historic aerial photographs and other information that indicated there were potential disturbance areas within portions of the Study Area. NYSDEC requested that Corning Incorporated perform preliminary characterization activities to assess the nature and extent of fill material that may be encountered within the Study Area.

In preparation for entering into an Order on Consent and Administrative Settlement (Order), Corning Incorporated has retained Weston Solutions, Inc. (WESTON®) to prepare this Study Area Characterization Work Plan (Work Plan) for the characterization activities to be conducted within the Study Area. It is Corning Incorporated's intention that this Work Plan, upon approval by NYSDEC, be incorporated into the Order to define the agreed scope of work.

1.1 STUDY AREA CHARACTERIZATION OBJECTIVES

The purpose of the characterization activities is to assess the nature and extent of fill that may be encountered within the Study Area. In accordance with the Order, Corning Incorporated is conducting a historic records search and review to establish a history of the Study Area and identify areas where fill may potentially have been placed. This records search includes a review of historic aerial photographs to identify areas where historic disturbances may have occurred. This Work Plan includes a summary of the initial historic records review and a plan for characterization activities based on the preliminary results of the historic records review. The characterization activities described herein are designed to assess the nature and extent of fill that

may be encountered within the Study Area and to develop data necessary for understanding the current conditions within the Study Area and associated potential exposure pathways.

The specific objectives of the Work Plan are as follows:

1. In areas where historic records indicate potential disturbances:

    a. assess the nature and extent of the potential disturbance area, and

    b. assess potential exposure pathways, in the event fill material is found.

2. In areas where historic records do not indicate potential disturbances, evaluate the potential presence of fill material.

1.2 ORGANIZATION OF THIS DOCUMENT

This Work Plan is organized into the following sections:

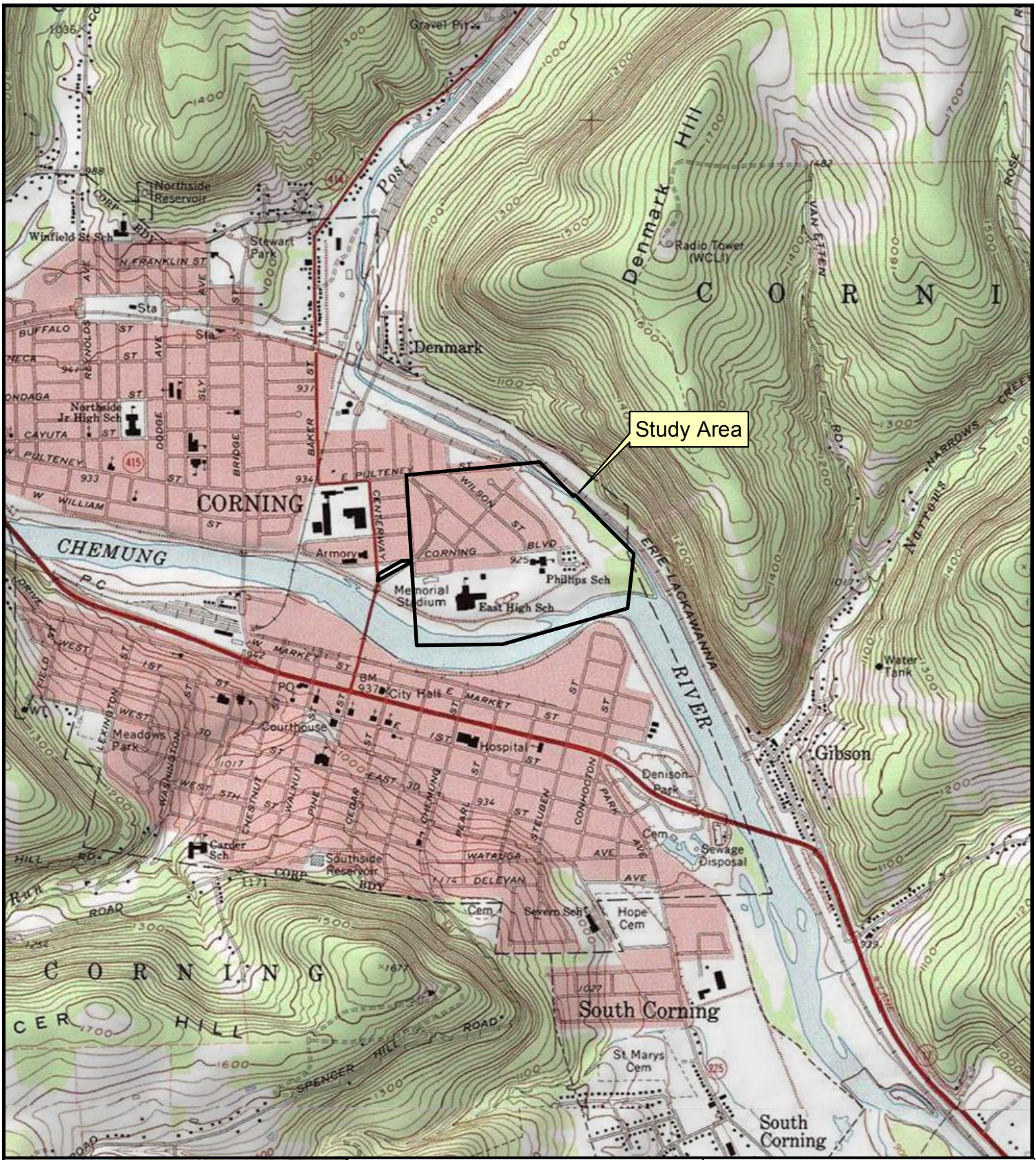
- **Section 1 - Introduction.** This section contains an introduction to the project and the objectives of the characterization activities.
- **Section 2 – Background.** This section contains a history of the Study Area and a summary of historic records reviewed to date.
- **Section 3 - Environmental Setting.** This section contains a brief description of the Study Area location, land use, topography and drainage, geology, hydrogeology, and ecological setting.
- **Section 4 – Characterization Activities.** This section contains a description of the characterization 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 characterization field work as well as the reporting schedule. Additionally, this section provides details about project logistics, including project controls, management and public 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 Work Plan.

SECTION 1

FIGURES



USGS 7.5 Minute Quadrangle Corning NY, 1971

Figure 1-1
Location of Study Area

Corning NY

Document Name: Site_Location.MXD

2. BACKGROUND

During construction activities as a part of the expansion of the Corning-Painted Post East High School located in the southern portion of the Study Area, fill materials that the School District described as containing ash, brick, and glass waste was encountered in the subsurface soils. As noted below, the land use within the Study Area has developed over time from farmland into a residential area and an area of schools and athletic fields. During development activities, "fill" material can commonly be used as sub-grade material for construction. In the context of this investigation, the term "fill" is used to refer to such sub-grade construction material and other material containing brick, ash, and glass waste. The term "fill" can also be used to describe cover that is brought in to support lawn and garden growth such as top soil and clay or sand. The term "cover" will be used in the context of this investigation to describe these materials and to distinguish them from other fill material.

To better understand the fill material encountered during the Corning-Painted Post East High School expansion project, the history of development in the Study Area has been investigated and historic aerial photographs have been reviewed. In addition, analytical data available from the Corning-Painted Post East High School expansion project have been reviewed to identify constituents of potential concern (COPCs), which will be the focus of the characterization work detailed in this Work Plan. A summary of the findings of these investigations/reviews are presented in the following subsections.

2.1 STUDY AREA HISTORY

The City of Corning has a long history of manufacturing, particularly in brick and glassmaking. Historical references indicate in the late 1800s and early 1900s, the City of Corning was home to a large brick manufacturer and more than sixty glass manufacturers (Dimitroff, 2001) (Sinclaire & Spillman, 1997). The most enduring of these enterprises is Corning Incorporated whose history dates back to 1868 when the Corning Flint Glass Works was established in Corning, New York. While the company has grown and expanded through the years and changed names to Corning Glass Works and currently to Corning Incorporated, the corporate headquarters has remained in Corning, New York.

In the late 1800s and early 1900s, coal was the primary fuel source in the Corning, New York area, and most of the local industries used coal to heat their furnaces. In the early 1900s natural gas was introduced to the region, and Corning Glass Works, along with many other industries and municipalities, converted their fuel sources to natural gas. The exact years during which this conversion occurred for Corning Glass Works are not known.

In addition, through a title search of property deeds, it was found that the Study Area was located on part of lands previously owned by Corning Homes, Inc., which had acquired these properties in 1920 from the heirs of the then-deceased founder of Corning Flint Glass Works. The deeds for these properties contained a condition that allowed Corning Glass Works to maintain structures, buildings and "ash dumps as now located" [on the properties]. Despite a thorough review of available historical documents and public records, Corning Incorporated has not, to date, located any maps or records that depict the potential location, if any, of the "ash dumps" as referenced in the deeds (i.e., as of 1920). In 1937, Corning Homes, Inc. sold portions of the properties with the same conditional language included, which has never been extinguished. Therefore, the Study Area has been established as the boundary of the 1937 deed, which is bounded by Pyrex Street on the west, E. Pulteney Street on the north, Post Creek on the east and the Chemung River on the south as illustrated on Figure 2-1.

2.2 AERIAL PHOTOGRAPH REVIEW

To evaluate the potential for fill material within the Study Area, WESTON, at the direction of Corning Incorporated, conducted a review of available historic aerial photographs for the Study Area. Features such as rivers, structures, bridges, and roads can be observed on aerial photographs, and a comparison of aerial photographs from different time frames can indicate development and other changes to the land use. In addition, areas of disturbance can be observed which could indicate activities such as preparation for construction, site grading, deposition, borrow, etc. They could also indicate areas of standing water, distressed vegetation, roads, trails, etc.

The following aerial photographs were reviewed for the Study Area:

- August 8, 1938
- May 8, 1942
- April 16, 1952
- July 11, 1955
- October 8, 1964
- March 30, 1968
- March/April 2011

In general, the aerial photographs indicate that the development of the residential area north of Corning Boulevard began prior to 1938 along Pyrex Street and Houghton Circle. It subsequently expanded in an easterly direction across farmlands until about 1964 when the residential area was mostly developed. In addition, the 1938 through 1964 aerial photographs appear to indicate there are potential disturbance areas in limited areas south of Corning Boulevard, and in one portion of the eastern end of Corning Boulevard, up to the earthen dikes along the Chemung River and Post Creek. By the 1968 aerial photograph, structures are observed south of Corning Boulevard in the areas of the Former Kent Phillips School and Corning-Painted Post East High School.

The potential disturbance areas observed on the 1938 through 1964 aerial photographs will be specifically investigated as part of the characterization work detailed in this Work Plan. The Study Area boundary has been established with the 1937 deed because the 1937 deed with the conditional language predated aerial photography.

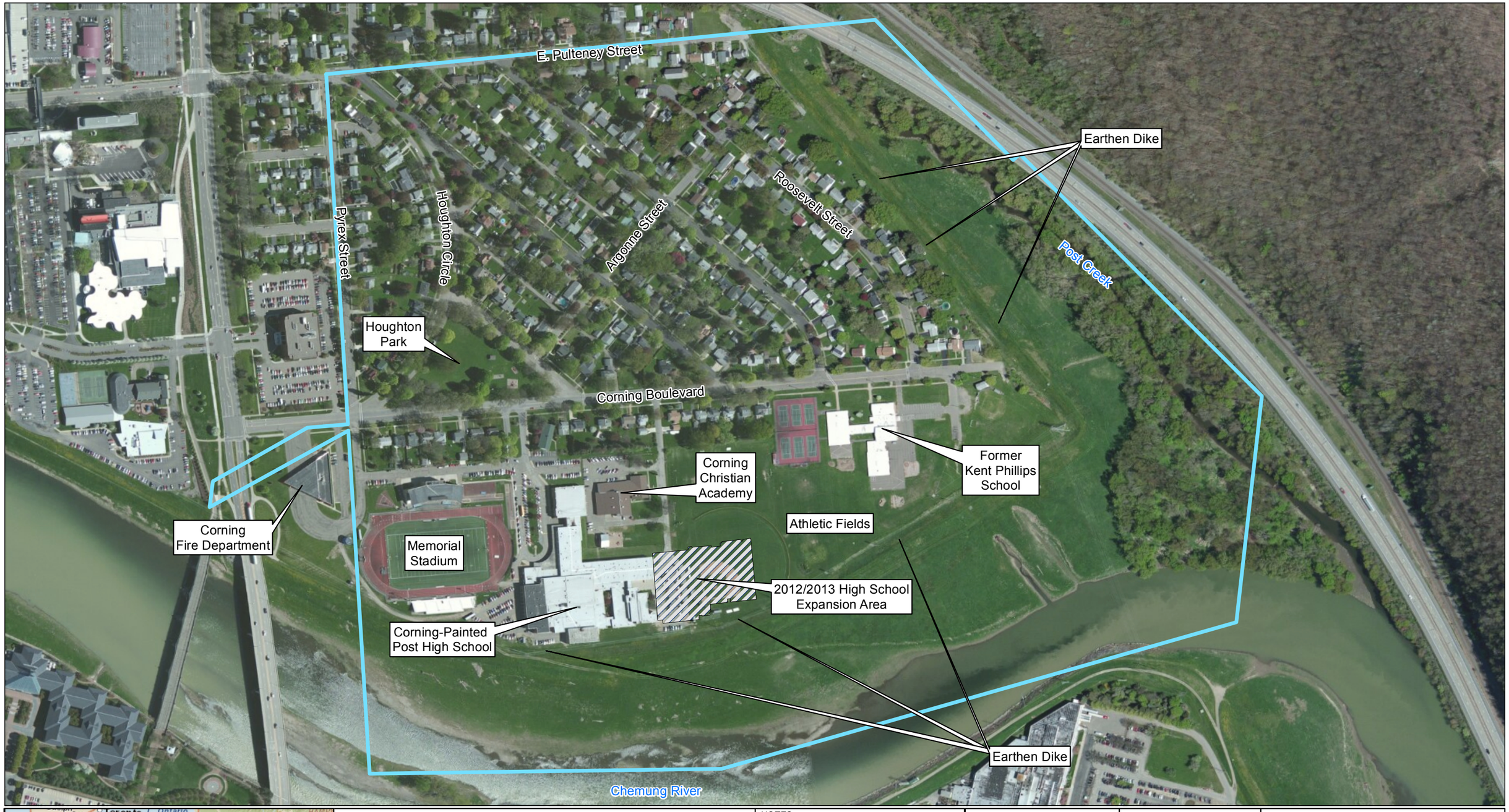
2.3 CONSTITUENTS OF POTENTIAL CONCERN

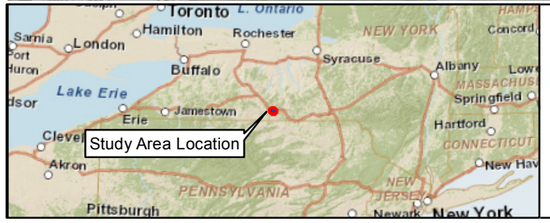
Based on the protocols the Corning-Painted Post School District had established for the construction activities at the high school, fill material that was encountered was excavated, segregated and sampled by the School District's consultant. The School District's consultant, in coordination with NYSDEC, based on the analytical results of the stockpiled material, disposed of approximately three quarters of the excavated material as non-hazardous solid waste and approximately one quarter of the excavated material as hazardous waste (NYSDEC, 2014).

A summary of the analytical results for the samples collected of the material excavated during the 2012 and 2013 expansion of the Corning-Painted Post East High School was supplied by the School District's consultant to the NYSDEC. A review of this data summary indicates there were over 200 samples of the excavated material collected and analyzed for various constituents. The majority of the constituents were either not detected or were reported at concentrations below the NYSDEC Residential Soil Cleanup Objectives (SCOs). The primary constituents which exceeded the Residential SCOs in the excavated materials were lead, cadmium, and arsenic; therefore, lead, cadmium and arsenic will be used as the COPCs for the characterization activities described in this Work Plan.

SECTION 2

FIGURES





Legend

Study Area Boundary Based on 1937 Quit Claim Deed

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye
Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

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Figure 2-1
Study Area

N

0 350 700 Feet

0 80 160 240 Meters

Document Name: Study_Area_Features.MXD

6/2/2014

3. ENVIRONMENTAL SETTING

3.1 LAND USE

The Study Area is generally bounded by Pyrex Street on the west, East Pulteney Street on the north, Post Creek on the east, and the Chemung River on the south. The Study Area consists of approximately 180 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 2-1). The Study Area includes the Corning-Painted Post East High School Property; the Corning Christian Academy Property; the Memorial Stadium Property; the Corning Firehouse Frontage Property; a residential area consisting of over 200 residences and a public park; 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 current industrial/commercial facilities are known to be located within the Study Area.

The City of Corning property classifications for the Study Area are illustrated on Figure 3-2. Currently, the portion of the Study Area south of Corning Boulevard and north of the earthen dike along the Chemung River is primarily used for educational purposes and is owned by the City of Corning, the Corning-Painted Post School District, and the Corning Christian Academy. North of the school area is predominately a residential area that consists of 217 individual properties. Of these properties, 210 are classified as single family residences, three are classified as multifamily residences or apartments, three are classified as residential-vacant land, and one is classified as a playground (i.e., Houghton Park). A list of the individual properties that comprise the Study Area, with the zoning, property classifications, and dates of construction is presented in Table 3-1.

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 is collected in storm water drains and generally flows south/southeast from the Study Area toward the Chemung River. Storm water is believed to be conveyed to the river through a storm drain(s) located in the southeast corner of the Study Area (as observed on aerial photographs). 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, portions of the Study Area, specifically, the flood control area, are located within both the Federal Emergency Management Agency (FEMA) 100-year and 500-year flood zones (FEMA, 2002).

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. In the vicinity of the Study Area, a low permeability, lacustrine silt and clay layer (approximately 10 feet thick) appears to be present about 30 feet below ground surface (ft bgs) (Miller, 1982). 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).

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 and has been modified by human influence to such a degree that the physical conformation of the substrate and the biological composition of the resident community is substantially different from the character of the substrate or community as it existed prior to human influence.

Within the residential area in the north of the Study Area, the ground cover is primarily mowed lawn with trees. The south-central portion of the Study Area is primarily used for educational purposes and the ground cover exists in the form of mowed lawn. Further south and to the east, a large expanse flood control area of mowed lawn habitat is present on the crest and slopes of the earthen dike.

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).

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.

SECTION 3

FIGURES

Legend

Study Area Boundary

Zoning

Public-Conservation

R1 - Low Density

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

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Figure 3-1
Zoning

N

0 350 700 Feet

0 80 160 240 Meters

Document Name: Zoning.MXD

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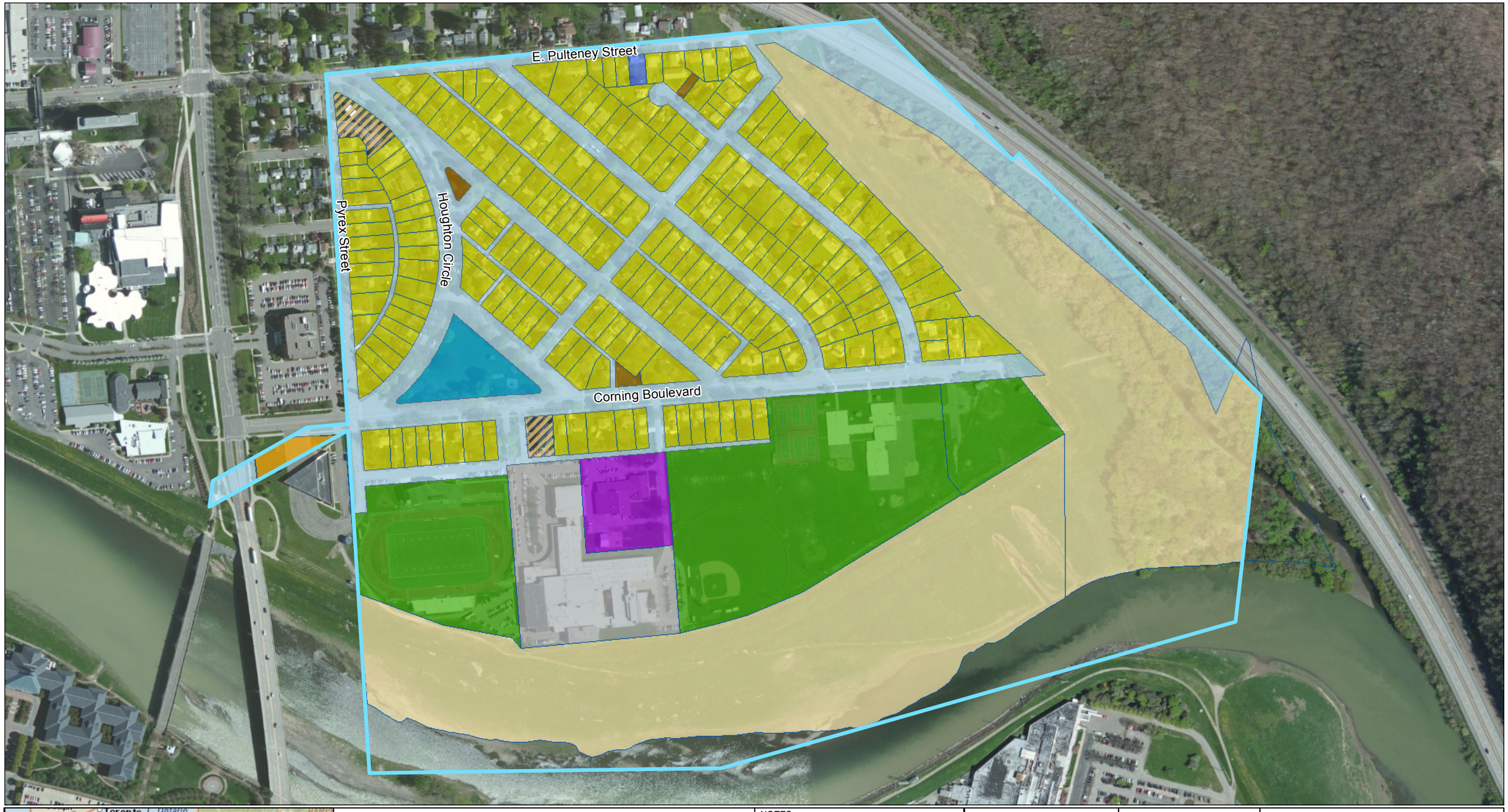


Legend

Study Area Boundary

Propety Classification

- 210 - Single Family Residence
- 220 - Multi Family Residence
- 311 - Residential - Vacant Land
- 411 - Apartment
- 591 - Playground
- 592 - Athletic Field
- 612 - School
- 615 - Educational Facility
- 622 - Fire/Police
- 821 - Flood Control
- Road

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye
Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

0 350 700 Feet

0 80 160 240 Meters

WESTON SOLUTIONS

Figure 3-2
Property Classification

Document Name: Property_Classification.MXD

6/2/2014

SECTION 3
TABLES

Table 3-1
Properties within the Boundaries of the Study Area
Corning, New York

Parcel ID	Address	Zoning	Property Classification	Year Built
318.05-03-029.000	67 Wilson St	R1	Single Family Residence	1952
318.05-03-032.000	236 E Pulteney St	R1	Single Family Residence	1950
318.05-03-033.000	240 E Pulteney St	R1	Single Family Residence	1973
318.05-03-028.000	65 Wilson St	R1	Single Family Residence	1974
318.05-03-030.000	232 E Pulteney St	R1	Single Family Residence	1950
318.05-03-031.000	234 E Pulteney St	R1	Single Family Residence	1945
318.05-01-006.000	61 Pershing St	R1	Single Family Residence	1956
318.05-01-004.000	4 Belleau St	R1	Single Family Residence	1960
318.05-01-005.000	65 Pershing St	R1	Single Family Residence	1926
318.05-01-010.000	47 Pershing St	R1	Single Family Residence	1950
318.05-01-011.000	45 Pershing St	R1	Single Family Residence	1973
317.08-01-039.000	Flood Control Area	PC	Flood Control Area	---
318.06-01-001.000	Flood Control Area	PC	Flood Control Area	---
318.05-01-003.000	6 Belleau St	R1	Single Family Residence	1928
318.05-01-009.000	49 Pershing St	R1	Single Family Residence	1926
318.05-01-013.000	33 Pershing St	R1	Single Family Residence	1927
318.05-01-014.000	29 Pershing St	R1	Single Family Residence	1955
318.05-01-018.000	29 Pershing St	R1	Single Family Residence	---
318.05-01-020.000	33 Sims Ave	R1	Single Family Residence	1999
318.05-01-021.000	31 Sims Ave	R1	Single Family Residence	1943
318.05-01-022.000	27 Sims Ave	R1	Single Family Residence	1930
318.05-01-024.000	19 Sims Ave	R1	Single Family Residence	1943
318.05-01-025.000	17 Sims Ave	R1	Single Family Residence	1940
318.05-01-015.000	25 Pershing St	R1	Single Family Residence	1926
318.05-01-017.000	75 Corning Blvd	R1	Single Family Residence	1940
318.05-01-023.000	21 Sims Ave	R1	Single Family Residence	1922
318.05-01-007.000	57 Pershing St	R1	Single Family Residence	1928
318.05-01-008.000	53 Pershing St	R1	Single Family Residence	1972
318.05-01-012.000	35 Pershing St	R1	Single Family Residence	1946
318.05-01-001.000	Pershing St (Memorial Stone)	R1	Residential - Vacant	---
318.05-01-019.000	65 Corning Blvd	R1	Single Family Residence	1939
318.05-01-035.000	18 Corning Blvd	R1	Single Family Residence	1930
318.05-01-045.000	74 Corning Blvd	R1	Single Family Residence	1928
318.05-01-047.000	82 Corning Blvd	R1	Single Family Residence	1930
318.05-01-048.000	84 Corning Blvd	R1	Single Family Residence	1929
318.05-01-026.000	15 Sims Ave	R1	Single Family Residence	1956
318.05-01-027.000	11 Sims Ave	R1	Single Family Residence	1940
318.05-01-028.000	42 Houghton Cir	R1	Single Family Residence	1940
318.05-01-029.000	40 Houghton Cir	R1	Single Family Residence	1910
318.05-01-039.000	26 Corning Blvd	R1	Single Family Residence	1974
318.05-01-044.000	66 Corning Blvd	R1	Single Family Residence	1935
318.05-01-050.000	100 Corning Blvd	R1	Single Family Residence	1945
318.05-01-068.000	202 Cantigny St (Stadium)	PC	Athletic Field	---
318.05-01-002.000	69 Pershing St	R1	Single Family Residence	1942
318.05-01-016.000	21 Pershing St	R1	Single Family Residence	1930
318.05-01-031.000	Houghton Park	R1	Playground	---
318.05-01-032.000	6 Corning Blvd	R1	Single Family Residence	1973
318.05-01-036.000	20 Corning Blvd	R1	Single Family Residence	1940
318.05-01-041.000	50 Corning Blvd	R1	Apartment	---
318.05-01-043.000	58 Corning Blvd	R1	Single Family Residence	1940
318.05-01-046.000	78 Corning Blvd	R1	Single Family Residence	1942
318.05-01-049.000	94 Corning Blvd	R1	Single Family Residence	1952

Table 3-1 (continued)
Properties within the Boundaries of the Study Area
Corning, New York

Parcel ID	Address	Zoning	Property Classification	Year Built
318.05-01-051.000	102 Corning Blvd	R1	Single Family Residence	1937
318.05-01-053.000	106 Corning Blvd	R1	Single Family Residence	1947
318.05-01-065.000	120 Corning Blvd	PC	School	---
318.05-01-054.000	126 Corning Blvd	PC	Athletic Field	---
318.05-01-067.000	201 Cantigny St	PC	School	---
318.05-01-066.000	11 Aisne St	R1	Educational Facility	---
318.05-02-046.000	67-99 Houghton Cir	R1	Apartment	---
318.05-02-050.000	59 Houghton Cir	R1	Single Family Residence	1952
318.05-02-052.000	53 Houghton Cir	R1	Single Family Residence	1920
318.05-02-054.000	47 Houghton Cir	R1	Single Family Residence	1952
318.05-01-033.000	10 Corning Blvd	R1	Single Family Residence	1919
318.05-01-034.000	14 Corning Blvd	R1	Single Family Residence	1920
318.05-01-037.000	22 Corning Blvd	R1	Single Family Residence	1950
318.05-01-040.000	40 Corning Blvd	R1	Single Family Residence	1950
318.05-01-042.000	54 Corning Blvd	R1	Single Family Residence	1954
318.05-01-052.000	104 Corning Blvd	R1	Single Family Residence	1938
318.05-02-053.000	49 Houghton Cir	R1	Single Family Residence	1951
318.05-02-057.000	33 Houghton Cir	R1	Single Family Residence	1973
318.05-02-059.000	21 Houghton Cir	R1	Single Family Residence	1972
318.05-02-060.000	19 Houghton Cir	R1	Single Family Residence	1937
318.05-02-062.000	13 Houghton Cir	R1	Single Family Residence	1945
318.05-02-063.000	11 Houghton Cir	R1	Single Family Residence	1951
318.05-02-064.000	9 Houghton Cir	R1	Single Family Residence	1935
318.05-02-066.000	24 Pyrex St	R1	Single Family Residence	1956
318.05-02-069.000	30 Pyrex St	R1	Single Family Residence	1929
318.05-02-071.000	34 Pyrex St	R1	Single Family Residence	1950
318.05-02-074.000	40 Pyrex St	R1	Single Family Residence	1920
318.05-02-077.000	46 Pyrex St	R1	Single Family Residence	1952
318.05-02-048.000	63 Houghton Cir	R1	Single Family Residence	1950
318.05-02-051.000	55 Houghton Cir	R1	Single Family Residence	1951
318.05-02-055.000	45 Houghton Cir	R1	Single Family Residence	1951
318.05-02-070.000	32 Pyrex St	R1	Single Family Residence	1950
318.05-02-076.000	44 Pyrex St	R1	Single Family Residence	1973
318.05-02-058.000	27 Houghton Cir	R1	Single Family Residence	1930
318.05-02-061.000	15 Houghton Cir	R1	Single Family Residence	1930
318.05-02-067.000	26 Pyrex St	R1	Single Family Residence	1972
318.05-02-049.000	61 Houghton Cir	R1	Single Family Residence	1955
318.05-02-056.000	39 Houghton Cir	R1	Single Family Residence	1928
318.05-02-068.000	28 Pyrex St	R1	Single Family Residence	1953
318.05-02-075.000	42 Pyrex St	R1	Single Family Residence	1952
318.05-02-078.000	48 Pyrex St	R1	Single Family Residence	1953
318.05-03-003.000	88 Pershing St	R1	Single Family Residence	1950
318.05-03-007.000	80 Pershing St	R1	Single Family Residence	1973
318.05-02-072.000	36 Pyrex St	R1	Single Family Residence	1950
318.05-02-073.000	38 Pyrex St	R1	Single Family Residence	1952
318.05-03-002.000	92 Pershing St	R1	Single Family Residence	1950
318.05-03-005.000	84 Pershing St	R1	Single Family Residence	1974
318.05-03-006.000	82 Pershing St	R1	Single Family Residence	1973
318.05-03-019.000	47 Wilson St	R1	Single Family Residence	1975
318.05-03-023.000	55 Wilson St	R1	Single Family Residence	1973
318.05-03-037.000	52 Wilson St	R1	Single Family Residence	1973

Table 3-1 (continued)
Properties within the Boundaries of the Study Area
Corning, New York

Parcel ID	Address	Zoning	Property Classification	Year Built
318.05-03-038.000	50 Wilson St	R1	Single Family Residence	1974
318.05-03-040.000	46 Wilson St	R1	Single Family Residence	---
318.05-03-042.000	42 Wilson St	R1	Single Family Residence	1950
318.05-03-045.000	111 Argonne St	R1	Single Family Residence	1957
318.05-03-046.000	6 Jackson Cir	R1	Single Family Residence	1940
318.05-03-049.000	12 Jackson Cir	R1	Single Family Residence	1948
318.05-03-054.000	262 E Pulteney St	R1	Single Family Residence	1958
318.05-03-055.000	260 E Pulteney St	R1	Single Family Residence	1927
318.05-03-008.000	66 Pershing St	R1	Single Family Residence	1938
318.05-03-010.000	62 Pershing St	R1	Single Family Residence	1948
318.05-03-014.000	50 Pershing St	R1	Single Family Residence	1975
318.05-03-022.000	53 Wilson St	R1	Single Family Residence	1973
318.05-03-056.000	264 E Pulteney St	R1	Single Family Residence	1960
318.05-03-058.000	268 E Pulteney St	R1	Single Family Residence	1960
318.05-04-002.000	34 Pershing St	R1	Single Family Residence	1940
318.05-04-003.000	30 Pershing St	R1	Single Family Residence	1925
318.05-04-006.000	18 Pershing St	R1	Single Family Residence	1928
318.05-03-052.000	252 E Pulteney St	R1	Single Family Residence	1973
318.05-03-053.000	258 E Pulteney St	R1	Single Family Residence	1927
318.05-03-060.000	121 Argonne St	R1	Single Family Residence	1973
318.05-03-063.000	115 Argonne St	R1	Single Family Residence	1956
318.05-03-009.000	64 Pershing St	R1	Single Family Residence	1973
318.05-03-011.000	60 Pershing St	R1	Single Family Residence	1943
318.05-03-013.000	54 Pershing St	R1	Single Family Residence	1945
318.05-03-015.000	42 Pershing St	R1	Single Family Residence	1925
318.05-03-020.000	49 Wilson St	R1	Single Family Residence	1948
318.05-03-026.000	61 Wilson St	R1	Single Family Residence	1974
318.05-03-036.000	54 Wilson St	R1	Single Family Residence	1973
318.05-03-043.000	34 Wilson St	R1	Single Family Residence	1950
318.05-03-001.000	94 Pershing St	R1	Single Family Residence	1946
318.05-03-004.000	86 Pershing St	R1	Single Family Residence	1950
318.05-03-012.000	58 Pershing St	R1	Single Family Residence	1940
318.05-03-016.000	99 Argonne St	R1	Single Family Residence	1973
318.05-03-018.000	45 Wilson St	R1	Single Family Residence	1975
318.05-03-025.000	59 Wilson St	R1	Single Family Residence	1974
318.05-03-034.000	244 E Pulteney St	R1	Single Family Residence	1952
318.05-03-035.000	56 Wilson St	R1	Single Family Residence	1973
318.05-03-064.000	113 Argonne St	R1	Single Family Residence	1960
318.05-03-065.000	7 Jackson Cir	R1	Single Family Residence	1946
318.05-03-066.000	17 Jackson Cir	R1	Residential - Vacant	---
318.05-04-013.000	7 Wilson St	R1	Single Family Residence	1948
318.05-04-017.000	15 Wilson St	R1	Single Family Residence	1947
318.05-04-007.000	14 Pershing St	R1	Single Family Residence	1937
318.05-04-009.000	8 Pershing St	R1	Single Family Residence	1969
318.05-04-014.000	9 Wilson St	R1	Single Family Residence	1947
318.05-04-015.000	11 Wilson St	R1	Single Family Residence	1948
318.05-04-019.000	19 Wilson St	R1	Single Family Residence	1947
318.05-04-022.000	25 Wilson St	R1	Single Family Residence	1947
318.05-04-023.000	27 Wilson St	R1	Single Family Residence	1947
318.05-04-029.000	28 Wilson St	R1	Single Family Residence	1947
318.05-04-032.000	22 Wilson St	R1	Single Family Residence	1951

Table 3-1 (continued)
Properties within the Boundaries of the Study Area
Corning, New York

Parcel ID	Address	Zoning	Property Classification	Year Built
318.05-04-038.000	10 Wilson St	R1	Single Family Residence	1942
318.05-04-040.000	6 Wilson St	R1	Single Family Residence	1947
318.05-04-042.000	115 Corning Blvd	R1	Single Family Residence	1950
318.05-04-046.000	7 Roosevelt St	R1	Single Family Residence	1974
318.05-03-044.000	109 Argonne St	R1	Single Family Residence	1977
318.05-03-047.000	8 Jackson Cir	R1	Single Family Residence	1956
318.05-03-048.000	10 Jackson Cir	R1	Single Family Residence	1945
318.05-03-050.000	248 E Pulteney St	R1	Single Family Residence	1952
318.05-03-057.000	266 E Pulteney St	R1	Single Family Residence	1960
318.05-03-059.000	270 E Pulteney St	R1	Single Family Residence	1959
318.05-04-004.000	26 Pershing St	R1	Single Family Residence	1925
318.05-04-021.000	23 Wilson St	R1	Single Family Residence	1949
318.05-04-024.000	29 Wilson St	R1	Single Family Residence	1948
318.05-04-028.000	30 Wilson St	R1	Single Family Residence	1947
318.05-04-031.000	24 Wilson St	R1	Single Family Residence	1948
318.05-04-034.000	18 Wilson St	R1	Single Family Residence	1947
318.05-04-036.000	14 Wilson St	R1	Single Family Residence	1946
318.05-03-041.000	44 Wilson St	R1	Single Family Residence	1951
318.05-03-051.000	250 E Pulteney St	R1	Single Family Residence	1955
318.05-04-039.000	8 Wilson St	R1	Single Family Residence	1977
318.05-04-043.000	117 Corning Blvd	R1	Single Family Residence	1975
318.05-04-052.000	21 Roosevelt St	R1	Single Family Residence	1973
318.05-03-062.000	117 Argonne St	R1	Single Family Residence	1974
318.05-04-001.000	36 Pershing St	R1	Single Family Residence	1937
318.05-04-005.000	22 Pershing St	R1	Single Family Residence	1927
318.05-04-008.000	10 Pershing St	R1	Single Family Residence	1925
318.05-04-010.000	105 Corning Blvd	R1	Single Family Residence	1973
318.05-04-011.000	107 Corning Blvd	R1	Single Family Residence	1947
318.05-04-049.000	15 Roosevelt St	R1	Single Family Residence	1976
318.05-04-050.000	17 Roosevelt St	R1	Single Family Residence	1977
318.05-04-051.000	19 Roosevelt St	R1	Single Family Residence	1973
318.05-04-055.000	27 Roosevelt St	R1	Single Family Residence	1973
318.05-04-020.000	21 Wilson St	R1	Single Family Residence	1947
318.05-04-026.000	104 Argonne St	R1	Single Family Residence	1978
318.05-04-030.000	26 Wilson St	R1	Single Family Residence	1948
318.05-04-056.000	33 Roosevelt St	R1	Single Family Residence	1973
318.05-04-058.000	37 Roosevelt St	R1	Single Family Residence	1973
318.05-04-061.000	108 Argonne St	R1	Single Family Residence	1977
318.05-04-063.000	44 Roosevelt St	R1	Single Family Residence	1951
318.05-04-069.000	30 Roosevelt St	R1	Single Family Residence	1978
318.05-04-074.000	18 Roosevelt St	R1	Single Family Residence	1976
318.05-04-080.000	8 Roosevelt St	R1	Single Family Residence	1973
318.05-04-054.000	25 Roosevelt St	R1	Single Family Residence	1973
318.05-04-064.000	42 Roosevelt St	R1	Single Family Residence	1976
318.05-04-067.000	36 Roosevelt St	R1	Single Family Residence	1973
318.05-04-068.000	34 Roosevelt St	R1	Single Family Residence	1973
318.05-04-070.000	28 Roosevelt St	R1	Single Family Residence	1974
318.05-04-072.000	24 Roosevelt St	R1	Single Family Residence	1976
318.05-04-073.000	20 Roosevelt St	R1	Single Family Residence	1973
318.05-04-084.000	127 Corning Blvd	R1	Single Family Residence	1973
318.05-04-012.000	109 Corning Blvd	R1	Single Family Residence	1946

Table 3-1 (continued)
Properties within the Boundaries of the Study Area
Corning, New York

Parcel ID	Address	Zoning	Property Classification	Year Built
318.05-04-016.000	13 Wilson St	R1	Single Family Residence	1947
318.05-04-018.000	17 Wilson St	R1	Single Family Residence	1973
318.05-04-025.000	98 Argonne St	R1	Single Family Residence	1973
318.05-04-033.000	20 Wilson St	R1	Single Family Residence	1947
318.05-04-035.000	16 Wilson St	R1	Single Family Residence	1947
318.05-04-037.000	12 Wilson St	R1	Single Family Residence	1947
318.05-04-057.000	35 Roosevelt St	R1	Single Family Residence	1975
318.05-04-060.000	39 Roosevelt St	R1	Single Family Residence	1975
318.05-04-062.000	46 Roosevelt St	R1	Single Family Residence	1973
318.05-04-076.000	16 Roosevelt St	R1	Single Family Residence	1975
318.05-04-078.000	12 Roosevelt St	R1	Single Family Residence	1973
318.05-04-081.000	4 Roosevelt St	R1	Single Family Residence	1974
318.05-04-041.000	4 Wilson St	R1	Single Family Residence	1976
318.05-04-045.000	121 Corning Blvd	R1	Single Family Residence	1975
318.05-04-048.000	11 Roosevelt St	R1	Single Family Residence	1974
318.05-04-066.000	38 Roosevelt St	R1	Single Family Residence	1973
318.05-04-079.000	10 Roosevelt St	R1	Single Family Residence	1975
318.05-04-082.000	123 Corning Blvd	R1	Single Family Residence	1976
318.05-04-083.000	125 Corning Blvd	R1	Single Family Residence	1975
318.05-02-1.2.000	1 Corning Blvd - Firehouse Frontage	PC	Fire/Police	---

Notes:
--- = Not available
R1 - low density zoning
PC - public/conservation zoning

4. CHARACTERIZATION ACTIVITIES

4.1 AREAS OF INVESTIGATION

The proposed characterization activities across the Study Area are focused primarily based upon current use and historic knowledge. For this Work Plan the Study Area has been divided into seven subareas, as follows: 1) Corning-Painted Post School District Property, 2) Corning Christian Academy Property, 3) Memorial Stadium Property, 4) Firehouse Frontage Property, 5) Residential Area at the Eastern End of Corning Boulevard, 6) Residential Area, and 7) Flood Control Area. The approximate limits of the subareas within the Study Area are identified on Figure 4-1.

1) Corning-Painted Post School District Property

The Corning-Painted Post School District Property consists of three parcels of contiguous land covering approximately 25 acres. All three parcels are owned by the Corning-Painted Post School District. According to City of Corning zoning information, all three parcels are zoned PC and classified as a School and Athletic Fields (see Figures 3-1 and 3-2).

Potential disturbance areas were identified in the central and eastern portions of the Corning-Painted Post School District Property during the review of historic aerial photographs. Construction activities associated with the expansion of the Corning-Painted Post East High School reportedly included excavation and removal of fill material and capping portions of the property with cover soil. The horizontal and vertical limits of fill material in this area were not determined during the school construction project.

2) Corning Christian Academy Property

The Corning Christian Academy Property is located on an approximate 2.6 acre parcel immediately north and east of the Corning-Painted Post East High School. This property is owned by the Corning Christian Academy, Inc. According to the City of Corning, the parcel is zoned PC, and classified as an Educational Facility (see Figures 3-1 and 3-2).

3) and 4) Memorial Stadium and Firehouse Frontage Properties

The Memorial Stadium property and a portion of the Firehouse Frontage property that lies within the 1937 deed footprint are located on two parcels covering approximately 7.9 acres in total. This land is owned by the City of Corning. According to the City of Corning, both parcels are zoned PC, and are classified as Athletic Field (Memorial Stadium Property) and Fire/Police (Firehouse Frontage Property), respectively (see Figures 3-1 and 3-2).

5) Residential Area at the Eastern End of Corning Boulevard

A small portion of the residential area located at the eastern end of Corning Boulevard has been defined as one of the areas of investigation. A potential disturbance area was identified in this area during the review of historic aerial photographs. A portion of this potential disturbance area was located north of Corning Boulevard, and a portion was located south of Corning Boulevard. The area south of Corning Boulevard is contained within the Corning-Painted Post School District Property area discussed above. The "Residential Area at the Eastern End of Corning Boulevard" refers to the portion of the potential disturbance area north of Corning Boulevard. The potential disturbance area north of Corning Boulevard covers portions of five parcels which are all zoned R1 by the City of Corning and classified as Single Family Residences (see Figures 3-1 and 3-2). For purposes of the characterization activities, the Residential Area at the Eastern End of Corning Boulevard includes the entire footprint of each of the affected five parcels, which encompass a total of approximately 2.5 acres.

6) Residential Area

The Residential Area of the Study Area shown on Figure 4-1 contains 212 individual parcels, totaling approximately 69 acres. The Residential Area consists of:

- An approximately 62-acre area north of Corning Boulevard. This area contains 191 parcels, and is bounded to the north by East Pulteney Street, to the south by Corning Boulevard, to the west by Pyrex Street, and to the east by the flood control dike along Post Creek. This area also includes one public park (Houghton Park).

- An approximately 7-acre area, consisting of 21 parcels, bounded to the north by Corning Boulevard, to the east by the tennis courts associated with the Corning-Painted Post East High School, to the west by Craumer Drive and to the south by Jacoby Boulevard and the Corning-Painted Post East High School.

All of the parcels within the defined Residential Area are zoned R1 by the City of Corning; 205 are classified as Single Family Residence, one is classified as a Multi Family Residence, three are classified as Residential-Vacant Land, two are classified as Apartment and one as Playground (i.e., Houghton Park) (see Figures 3-1 and 3-2).

7) Flood Control Area

The Flood Control Area is confined to the areas along the eastern and southern boundaries of the Study Area. The Flood Control Area is the area between the Residential Area and the banks of Post Creek as well as the area between the Corning-Painted Post School District Property and the Chemung River including the earthen dikes. The Flood Control Area property is owned by the City of Corning and it covers two parcels consisting of approximately 73 acres. The Flood Control Area is zoned PC and classified as Flood Control (see Figures 3-1 and 3-2).

4.2 FIELD INVESTIGATION METHODOLOGIES

The subsections below describe the field investigation methodologies to be utilized for the characterization activities at the Study Area. The proposed methodologies may be adjusted in the field based upon a variety of factors including field conditions, selected subcontractor equipment and other necessary adjustments. The NYSDEC will be notified of any proposed significant changes or deviations from the approved Work Plan (including any proposed use of investigation methodologies other than those described below) and NYSDEC approval will be obtained prior to implementation. Minor field adjustments or the addition of sampling locations that do not affect the project objectives will be discussed verbally with the NYSDEC project manager, confirmed by subsequent email and/or documented in the field notes, and ultimately noted in the investigation summary report.

Planned soil characterization activities include a combination of soil boring and surface soil sampling. Characterization activities may also include the installation of shallow groundwater

monitoring wells. The number of sampling locations, specific to each designated subarea, is described in the subsequent subsections. Final locations will be established based on utility clearance, accessibility, and discussions with property owners/lessees. Standard Operating Procedures (SOPs) for sample collection, handling and shipment are provided in Appendix D.

4.2.1 Written Access Consent

Property within the Study Area is not owned by or under the control of Corning Incorporated or the NYSDEC. Therefore, written access consent between Corning Incorporated and individual property owners will be needed prior to the field investigation. It is expected that some sampling locations may need to be modified as a result of access issues. Corning Incorporated will provide two separate time periods for the residential homeowners to consent to access.

To ensure that the field work can be conducted expeditiously, safely, and with minimal impact to the community, field work will be staged to the extent possible to investigate as many locations during the first investigation period as possible. Provision will be made for a second investigation period approximately 60 days later to pick up any subsequently received consent agreements. Thus, written access consent will need to be pursued and obtained in a timely manner.

Prior to performing work at each property, and after obtaining written access consent, the owners will be notified of pending activities on their properties.

4.2.2 Geophysical Investigation

Non-intrusive subsurface scans will be conducted using a combination of geophysical methods to assist in identifying subsurface stratigraphic details. Electromagnetic (EM) terrain conductivity and ground penetrating radar (GPR) will be employed to provide information to assist with the identification of the extent of fill material that may be encountered in the Study Area. The subsurface signals from fill should differ from native soil, and this will be verified in the field. If verified, the sub-surface geophysical scans can be used to help identify areas of potential fill, which could influence subsequent characterization activities. Both instruments will be interfaced with a Global Positioning System (GPS) to geo-reference the data.

The first stage of geophysical investigation is the EM survey. An EM survey will be conducted using a terrain conductivity meter. The instrument measures apparent conductivity in units of millisiemens per meter (mS/m) in materials with conductivities typically ranging up to 1,000 mS/m.

The EM unit will be operated in a "continuous" mode along pre-established parallel survey lines spaced at approximately 10-foot intervals. Measurements will be recorded at approximate 1 to 2.5-foot intervals as the operator traverses the grid. Measurements will be digitally recorded and stored in memory in a data logger.

At the completion of the EM survey, data stored in the data logger will be downloaded to a field computer for review by qualified WESTON personnel. The computer-generated output files will be reviewed to identify potential subsurface signals that differ in appearance, thus indicating potential fill material.

Following the EM survey, a follow up GPR survey will be conducted to provide information to enhance the resolution and depth of specific major anomalies/boundaries identified by the EM survey. Typically, GPR surveying will be performed using a Geophysical Survey Systems, Inc. GPR System 3000 radar unit. The GPR System 3000 unit consists of a control/display unit, mainframe/data storage unit, and 300- or 500-megahertz antenna. Surveying will be accomplished as follows:

- The GPR survey will consist of a series of transects crossing apparent boundaries of any major subsurface anomalies identified by the EM survey.
- The product of the GPR survey will be a series of real time radar profiles.
- Preliminary interpretation of the GPR profiles and EM field data will be done in the field to help mark potential areas of fill material.

4.2.3 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. Soil boring locations will be recorded using a hand-held GPS with sub-meter accuracy.

At each Geoprobe boring location, soil sampling will be conducted on a continuous basis (if possible) in 2-foot intervals. Retrieved soil samples will be examined in the field for physical description by a qualified WESTON geologist and screened using a photoionization detector (PID). Fill will be identified in the field as any soil containing non-native material. All Geoprobe rods and associated drilling equipment will be cleaned between boring locations using the procedures described in Appendix D.

Where a 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. 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, 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.). 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, or disposable equipment (e.g., scoops, plastic blending trays) will be used.

Soil samples and appropriate quality control (QC) samples (e.g., duplicate samples) will be collected from the sampling spoon cores, 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 or asphalt). 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 a designated location outside the Study Area approved by NYSDEC pending proper disposal.

4.2.4 Surface and Shallow Soil Sampling

Surface soil samples will be collected for analysis from 0 to 2 inches below ground surface (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. 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. Surface and shallow soil samples will be homogenized (for analyses other than volatiles) and placed directly into appropriate sample containers. The soil will be described as appropriate noting the color, moisture content, texture, layering, evidence of disturbance (foreign debris), and the distribution/abundance of roots. Prior to sample collection, gross vegetative matter will be removed (i.e., sod layer).

Generally, samples will be preferentially positioned to focus on play grounds, walkways, traffic and garden areas, bare soil areas, or near visibly disturbed soils, to provide general coverage of non-impervious surfaces at each property. Surface soil and shallow soil sample locations will be recorded 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 or disposable

equipment (e.g., scoops, plastic blending trays) will 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 a designated location outside the Study Area approved by NYSDEC 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.

4.2.5 Groundwater

Groundwater monitoring wells will be installed in areas as discussed in the following Subsections using hollow-stem auger drilling techniques. At the drilling location, the hollow-stem augers will be extended from ground surface to approximately 10 feet below the water table (estimated to be 20-25 ft bgs). Final well depths will be determined in the field based upon the estimated depth to the water table as evident from the drill cuttings. Soil samples will be collected with a split-spoon at two-foot intervals during drilling for physical description by a qualified WESTON geologist and screened with a PID.

Upon reaching the final depth, the well components will be placed within the augers. Well components are anticipated to consist of 10 feet of 2-inch diameter, 0.010-inch slot polyvinyl chloride (PVC) screen and the appropriate length of PVC riser piping. Following placement of the well components in the hollow-stem augers, a filter pack consisting of clean quartz sand will be placed from the bottom of the well screen to approximately 2 feet above the top of the well screen. A bentonite seal (approximately 2 feet thick) will then be placed above the filter pack. The remainder of the annular space above the bentonite seal will be backfilled with a cement/bentonite grout mixture to ground surface using a tremie rod. Each well will be completed flush to ground or with a minimum one-foot stickup depending on the agreement reached with the property owner. Locks will be placed on all wells.

After each new monitoring well is installed, it will be developed using a submersible pump to surge and pump the well until sediment production is negligible. New monitoring wells will be

allowed to set for at least 24 hours prior to development. Following well development, the new monitoring wells will be horizontally and vertically (top of casing and ground surface) located by a surveyor licensed in the State of New York. IDW (i.e., development water) from this investigation will be collected and contained in sealed containers (e.g., drums or other appropriate containers) and staged in a secondary containment area at a designated location outside the Study Area approved by NYSDEC pending proper disposal.

Two rounds of groundwater samples for laboratory analysis will be collected from the new monitoring wells. Monitoring well sampling will be performed no sooner than two weeks from completion of new well development. The second round of groundwater samples will be conducted three to six months following the collection of the first round of sampling. Monitoring well samples will be collected utilizing low-flow, low turbidity sampling procedures. Additional information regarding sampling technique is included in the SOPs, which are provided in Appendix D.

Groundwater samples and appropriate QC samples will be placed in iced coolers and shipped with completed chain-of-custody documentation to TestAmerica for analysis.

Prior to collection of groundwater samples for analysis, one round of water level measurements will be collected from all Study Area monitoring wells. A clean electronic water-level indicator will be lowered into each well to determine depth to water, and the top of casing elevation will be used to calculate groundwater level elevation above mean sea level.

4.2.6 Analytics

A summary of sampling, including the number of samples and anticipated analysis is provided in Table 4-1. All samples collected during the characterization program will be analyzed for the COPCs: arsenic, cadmium, and lead.

In addition to the COPCs, 20% of all soil and groundwater samples will be analyzed for an expanded analytical list; which includes total Target Analyte List (TAL) metals, Toxicity Characteristic Leaching Procedure (TCLP) Resource Conservation Recovery Act (RCRA) metals (soils only), total petroleum hydrocarbons (TPH), Target Compound List (TCL)

polychlorinated bi-phenyls (PCBs), and TCL semi-volatile organic compounds (SVOCs) (expanded list). Analysis for volatile organic compounds (VOCs) will also be conducted for soil boring samples where hand-held PID readings are above five parts per million above background levels.

Detailed descriptions of the sampling approach and rationale are included in Section 4.3 of this 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.

4.2.7 Quality Assurance / Quality Control

To ensure quality throughout the project, the involvement of trained and experienced personnel will be utilized, and proven operating procedures and analytical methods for sample collection, preservation, analysis, and documentation will be followed.

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 obtained 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:

- Trip Blanks: These samples are applicable to VOC analyses, and therefore, will accompany each of the sample shipments that are analyzed for VOCs. They will be prepared by the laboratory from deionized water, and will accompany the project samples through all custody changes to provide information regarding possible contamination introduced during the sample handling process.

- 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 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 5.2.2.

4.2.8 Survey Activities

All 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 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.

4.2.9 Waste Handling

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.

All non-dedicated sampling and monitoring equipment will be decontaminated by washing with phosphate-free detergent and rinsing with distilled water, or through other manufacturer approved decontamination methods. All rinse water, well development water, and purge water will be containerized and properly disposed by Corning Incorporated in accordance with applicable requirements.

4.3 INVESTIGATION ACTIVITIES

The following subsections describe the planned investigation activities for each of the seven subareas of the Study Area. The planned investigation activities for each area are depicted on Figures 4-2 through 4-9; however, the final locations will be determined based upon written access consent and field conditions. Furthermore, the proposed number of sample locations and associated analysis described herein and summarized in Tables 4-1 and 4-2 may be adjusted in the field based upon actual conditions and findings.

4.3.1 Corning-Painted Post School District Property

As described in Section 2, a review of aerial photographs indicated potential disturbance areas within the current boundary of the Corning-Painted Post School District Property. In addition, during recent construction activities as a part of the expansion of the high school, fill material that the School District described as containing ash, brick and glass waste was encountered in the subsurface soils. Based on this, the key investigation activities at the Corning-Painted Post School Property are: 1) soil cover evaluation to measure the thickness of the soil cover placed for the new athletic fields, 2) surface and shallow soil sampling for chemical analysis in areas where a soil cover was not recently placed, 3) a soil boring program to assess the nature and extent of fill material, and 4) a groundwater sampling program to assess potential impacts to the local groundwater. The proposed number of samples, including proposed analysis and QC samples, is summarized in Table 4-1. The investigation program has taken into consideration that most of the Corning-Painted Post School District Property is covered by either buildings, asphalt or a soil cover recently placed on the new athletic fields located on the eastern portion of the property.

4.3.1.1 Records Review

All records regarding the school expansion project provided by the Corning-Painted Post School District in response to a Freedom of Information Law (FOIL) request submitted on behalf of Corning Incorporated have been reviewed by WESTON prior to implementing fieldwork. Documentation regarding the source(s) of fill material used to construct the cover for the new athletic fields and analytical data of the cover material to the extent available has been reviewed. In the event that available records are determined to be satisfactory by the NYSDEC, certain

tasks described below (e.g. verification of soil cover thickness) may be modified as appropriate with NYSDEC approval.

4.3.1.2 Geophysical Investigation

A geophysical investigation will be conducted in the eastern portion of the Corning-Painted Post School District Property where potential disturbance areas were observed on historic aerial photographs. The purpose of the geophysical investigation will be to identify any subsurface features in an attempt to define boundaries of subsurface, non-native material.

4.3.1.3 Soil Cover Evaluation

An extensive soil cover evaluation program will be performed to verify the extent and thickness of the soil cover in the new athletic fields. As depicted on Figure 4-2, locations will be a based on an approximately 100-foot x 100-foot grid system across the open space in the school area. Locations will generally be placed at or near the grid nodes; however, in some cases the locations will be moved to open spaces. The grid will be laid out across the area of investigation and those locations that fall within the footprint of the buildings or other impervious surfaces will be excluded. At those locations which fall within the soil cover area in the new athletic fields, the thickness of the soil cover will be determined by collecting soil cores using hand-held steel soil augers or similar devices and measuring the cover thickness in the recovered soil core. While use of a hand auger is anticipated, if field conditions make sampling difficult, a small track-mounted Geoprobe rig could be used to collect the soil core within the 0 to 2 ft bgs interval or to confirm soil cover thickness.

4.3.1.4 Surface and Shallow Soil Sampling

For all areas of existing soil (outside of the new soil cover area), surface and shallow soil samples will be collected for chemical analysis. Surface soil samples will be collected from the 0 to 2 in bgs interval excluding the vegetative cover or sod layer, and shallow soil samples will be collected from the 2 in bgs to 2 ft bgs interval excluding the vegetative cover or sod layer. While use of a hand auger is anticipated, if field conditions make sampling difficult, a small track-mounted Geoprobe rig could be used to collect the soil samples within the 2 in bgs to 2 ft bgs

sampling interval. It should be noted that the grass and root layer 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. No samples will be collected on the earthen dike or within the floodplain area. All samples will be analyzed for COPCs and 20% of the samples will be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized in Table 4-1.

4.3.1.5 Soil Borings

In addition to the soil cover evaluation and surface and shallow soil sampling discussed in Sections 4.3.1.3 and 4.3.1.4 above, approximately 14 soil borings will be installed to characterize subsurface conditions in the Corning-Painted Post School District Property. The preliminary layout of these 14 soil borings is shown on Figure 4-2; however, the locations of these borings may be adjusted, with verbal approval by the NYSDEC Project Manager, followed by email confirmation and/or documentation in the field notes, based on field site conditions, access issues and/or results of the geophysical survey performed in this area. A majority of the soil borings are concentrated in the eastern portion of the investigation area where apparent disturbance areas were observed on certain historic aerial photographs. No soil borings are planned for areas underneath existing buildings. The 14 soil borings will be advanced via Geoprobe or hollow-stem auger to approximately 15 ft bgs or deeper as needed to reach native material.

If fill material is encountered while drilling the borings, detailed logs will be recorded, and two to three soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs, excluding the sod layer) or from the 2-foot interval immediately beneath the defined, newly placed, soil cover; one from the zone of observed fill material; and one from the native material immediately beneath the fill material.

In borings where no fill material is encountered in the boring and a new soil cover exists (e.g. playing field areas), two soil samples will be collected per boring; one from the 2-foot interval immediately beneath the defined soil cover and one sample of the native material at depth.

In borings where no fill material is encountered in the boring and where a soil cover was not recently placed, two soil samples will be collected per boring; one from soil in the top 2-foot interval where present (i.e. 0 to 2 ft bgs, excluding the sod layer) and one sample of the native material at depth.

All samples from soil boring locations will be analyzed for COPCs and 20% of the samples will also be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.1.6 Groundwater

A groundwater investigation is also planned for the Corning-Painted Post School District Property. It is anticipated that three groundwater monitoring wells will be installed in the vicinity of the school area. Two rounds of groundwater samples will be collected and analyzed for COPCs. Samples collected from one well during each round of groundwater sampling will be analyzed for the expanded list of parameters. In addition, groundwater samples for COPCs will be collected from existing wells in the school area, dependent upon prior approval from the well owner.

4.3.2 Corning Christian Academy Property

The characterization activities planned in the Corning Christian Academy Property include a combination of surface and shallow soil sampling and soil boring activities. The proposed number of samples, including the proposed analyses and QC samples is summarized in Table 4-1. Additional sampling detail is included in the QAPP provided in Appendix C.

4.3.2.1 Surface and Shallow Sampling

The surface and shallow soil grid system in the Corning-Painted Post School District Property will be expanded across the Corning Christian Academy Property and surface and shallow soil samples will be collected generally at or near the grid nodes; however, in some cases the locations will be moved to open spaces, with verbal approval by the NYSDEC Project Manager, followed by email confirmation and/or documentation in the field notes. Additional sample locations have been added in areas of playgrounds, etc. Surface soil samples will be collected

from the 0 to 2 in bgs interval excluding the mulch, vegetative cover or sod layer, and shallow soil samples will be collected from the 2 in bgs to 2 ft bgs interval excluding the mulch, vegetative cover, or sod layer. While use of a hand auger is anticipated, if field conditions make sampling difficult, a small track-mounted Geoprobe rig could be used to collect samples within the 2 in bgs to 2 ft bgs sampling interval. Surface soil sampling locations will be adjusted in the field as needed, with verbal approval by the NYSDEC Project Manager, followed by email confirmation and/or documentation in the field notes; no surface soil sample will be collected at locations with an impervious surface. It should be noted that the grass and root layer 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.

Additionally, the Corning Christian Academy Property contains a playground area that is covered by wood chips/mulch. In this playground area, the thickness of the mulch will be measured in approximately five locations and two shallow samples of the soil below the mulch will be collected for analysis. All surface and shallow soil samples will be analyzed for COPCs and 20% of the samples will be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized in Table 4-1.

4.3.2.2 Soil Borings

In addition to the surface and shallow soil sampling, two soil borings will be installed to characterize subsurface conditions in the Corning Christian Academy Property. The soil borings will be advanced via Geoprobe or hollow-stem auger to approximately 15 ft bgs or deeper as needed to reach native material. The preliminary layout of these soil borings is shown on Figure 4-3; however, the locations of these borings may be adjusted based on field conditions and access issues.

If fill material is encountered while drilling the borings, detailed logs will be recorded, and three soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs excluding the sod layer), one from the zone of observed fill material, and one from the native material immediately beneath the fill material. In borings where no fill material is encountered in the boring, two soil samples will be collected per boring; one from the

soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs, excluding the sod layer), and one sample of the native material at depth. All samples from soil boring locations will be analyzed for COPCs and 20% of the samples will also be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.3 Memorial Stadium and Firehouse Frontage Properties

The characterization activities planned in the Memorial Stadium and Firehouse Frontage Properties include a combination of surface and shallow soil sampling and soil boring activities. The proposed number of samples, including the proposed analyses and QC samples is summarized in Table 4-1. Additional sampling detail is included in the QAPP provided in Appendix C.

4.3.3.1 Surface and Shallow Sampling

The surface and shallow soil grid system in the Corning-Painted Post School District Property will be expanded across the Memorial Stadium Property and Firehouse Frontage Property. Surface and shallow soil samples will be collected at or near the grid nodes; however, in some cases the locations will be moved to open spaces. Surface soil samples will be collected from the 0 to 2 in bgs interval, excluding the vegetative cover or sod layer, and shallow soil samples will be collected from the 2 in bgs to 2 ft bgs interval excluding the vegetative cover or sod layer. While use of a hand auger is anticipated, if field conditions make sampling difficult, a small track-mounted Geoprobe rig could be used to collect shallow samples within the 2 in bgs to 2 ft bgs sampling interval. Sampling locations will be adjusted in the field as needed, with the verbal approval of the NYSDEC Project Manager, followed by email confirmation and/or documentation in the field notes; no surface soil sample will be collected at locations with an impervious surface. It should be noted that the grass and root layer 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. All surface and shallow soil samples will be analyzed for COPCs and 20% of the samples will be analyzed for the expanded list of

parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.3.2 Soil Boring

In addition to the surface and shallow soil sampling, three soil borings will be installed in each of the Memorial Stadium Property and Corning Firehouse Frontage Property. The soil borings will be advanced via Geoprobe or hollow-stem auger to approximately 15 ft bgs or deeper as needed to reach native material. The preliminary layout of these soil borings is shown on Figures 4-4 and 4-5; however, the locations of these borings may be adjusted based on field conditions and access issues.

If fill material is encountered while drilling the borings, detailed logs will be recorded, and three soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs excluding the sod layer), one from the zone of observed fill material, and one from the native material immediately beneath the fill material. In borings where no fill material is encountered in the boring, two soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs, excluding the sod layer), and one sample of the native material at depth. All samples from soil boring locations will be analyzed for COPCs and 20% of the samples will also be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.4 Residential Area at the Eastern End of Corning Boulevard

The characterization activities planned in the Residential Area at the Eastern End of Corning Boulevard include a combination of surface soil sampling and soil boring activities. The proposed number of samples, including the proposed analyses and QC samples is summarized in Table 4-1. Additional sampling detail is included in the QAPP provided in Appendix C.

4.3.4.1 Surface Soil

Prior to the installation of each soil boring in the Residential Area at the Eastern End of Corning Boulevard, one surface soil sample (0 to 2 in bgs, excluding the sod layer) will be collected at the

soil boring location. Two surface soil samples are proposed at each of the residential properties in this area as shown on Figure 4-6. One surface soil sample per property will be analyzed for the expanded list of parameters. It should be noted that the grass and root layer 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.

4.3.4.2 Soil Boring

Eleven soil borings will be installed, approximately two borings at each of the five parcels with in this area, as shown on Figure 4-6. The planned locations of the soil borings are approximate and final boring locations will be determined based in part on utility clearance and accessibility. Furthermore, the proposed boring locations are contingent upon the respective property owners providing written access consent to installing them on their property. The soil borings will be advanced via Geoprobe or hollow-stem auger to approximately 15 ft bgs or deeper as needed to reach native material.

If fill material is encountered while drilling the borings, detailed logs will be recorded, and three soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs excluding the sod layer), one from the zone of observed fill material, and one from the native material immediately beneath the fill material. In borings where no fill material is encountered in the boring, two soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs, excluding the sod layer), and one sample of the native material at depth. All samples from soil boring locations will be analyzed for COPCs and 20% of the samples will also be analyzed for the expanded list of parameters, at a minimum of one boring per property. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.4.3 Groundwater

As needed, groundwater monitoring may be performed in the Residential Area at the Eastern End of Corning Boulevard. The need for groundwater monitoring will be predicated on the analytical results from the soil sampling and may be combined with groundwater monitoring performed in other Study Area subareas.

If groundwater monitoring is conducted, groundwater monitoring wells will be installed, and two rounds of groundwater samples will be collected and analyzed for COPCs. Samples collected from one well during each round of groundwater sampling will also be analyzed for the expanded list of parameters.

4.3.5 Residential Area

The characterization activities planned in the Residential Area include a field reconnaissance survey, soil boring activities, and surface soil sampling. The proposed number of samples, including the proposed analyses and QC samples is summarized in Table 4-1. Additional sampling detail is included in the QAPP provided in Appendix C.

4.3.5.1 Field Reconnaissance

During activities in the Residential Area, WESTON personnel will perform field reconnaissance throughout the neighborhood and obtain pertinent information on properties to support a thorough evaluation of the Residential Area. During the reconnaissance, information such as the type and number of structures, areas of impervious surfaces, presence or absence of gardens, swing sets, etc. will be collected. WESTON personnel will conduct the field reconnaissance from public areas (streets, sidewalks, alleys, etc.) and will not enter private properties unless written access consent is provided to do so.

4.3.5.2 Soil Borings

Approximately 24 soil borings will be installed throughout the Residential Area. The soil borings are planned to be installed in the City of Corning right-of-way areas in the Residential Area at locations identified on Figure 4-7. These locations are approximate and final boring locations will be determined, with the verbal approval of the NYSDEC Project Manager, followed by email confirmation and/or documentation in the field notes, based in part on avoidance of backfill from historical utility or other road work, obtaining utility clearances and accessibility for equipment. Furthermore, the borings are contingent upon the City of Corning providing access to the rights-of-way and utility clearances. The soil borings will be advanced via

Geoprobe or hollow-stem auger to approximately 15 ft bgs or deeper as needed to reach native material.

If fill material is encountered while drilling the borings, detailed logs will be recorded, and two to three soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs excluding the sod layer), one from the zone of observed fill material, and one from the native material immediately beneath the fill material. In borings where no historic fill material is encountered in the boring, two soil samples will be collected per boring; one from the soil in the top 2-foot interval where present (i.e., 0 to 2 ft bgs, excluding the sod layer), and one sample of the native material at depth. All samples from soil boring locations will be analyzed for COPCs and 20% of the samples will also be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

4.3.5.3 Surface Soil Sampling

Along with the soil borings in the Residential Area, an extensive surface soil sampling program will be performed to assess potential exposure pathways within the Study Area. The surface soil campaign will require written access consent to be obtained from each individual property owner; therefore, the surface soil sampling campaign will be performed in a phased approach based on the ability to obtain written access consent.

The initial phase of surface soil sampling in the Residential Area is planned to be conducted approximately 60 days after the public notice is issued to the residents. The initial phase will only be conducted when written access consent is obtained from a significant quantity of the properties, with a representative spatial distribution throughout the Residential Area. A second phase of sampling will be conducted approximately 60 days after the initial phase depending on the ability to obtain additional written access consent.

Only after written access consent agreements are signed will sampling be performed on the individual properties. This sampling will involve three to four surface soil samples collected from the 0 to 2 in bgs interval, excluding the sod layer, on each property and analyzed for

COPCs. Unless modified by NYSDEC based on the soil boring data obtained from the adjacent right-of-way areas, one surface soil sample per property will also be analyzed for the expanded list of parameters. The locations of the surface soil samples will be determined in the field in locations biased toward entrance areas, swing sets, gardens, etc. It should be noted that 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.

If Corning Incorporated submits a request and provides NYSDEC with preliminary data generated from soil borings completed in the rights-of-way of the Residential Area within 45 days after the public notice is issued to the residents and a rationale for modifications to the expanded analytical list, NYSDEC will consider potential modifications to the expanded analytical list to be performed on surface soil samples collected from residential properties.

Due to the larger size of Houghton Park located within the Residential Area, additional surface soil sampling will be conducted in the park. A total of ten surface soil sample locations depicted on Figure 4-8 will be sampled from the 0 to 2 in bgs interval, excluding the sod layer. Additionally, in the three playground areas that are believed to be covered by wood chips/mulch, the thickness of the mulch will be measured in approximately five locations (each corner and center) and one sample of the material from the 0 to 2 in interval immediately beneath the mulch will be collected from each area for analysis. All samples will be analyzed for COPCs and 20% of the samples will be analyzed for the expanded list of parameters. The anticipated number of samples, including proposed analysis and QC samples is summarized on Table 4-1.

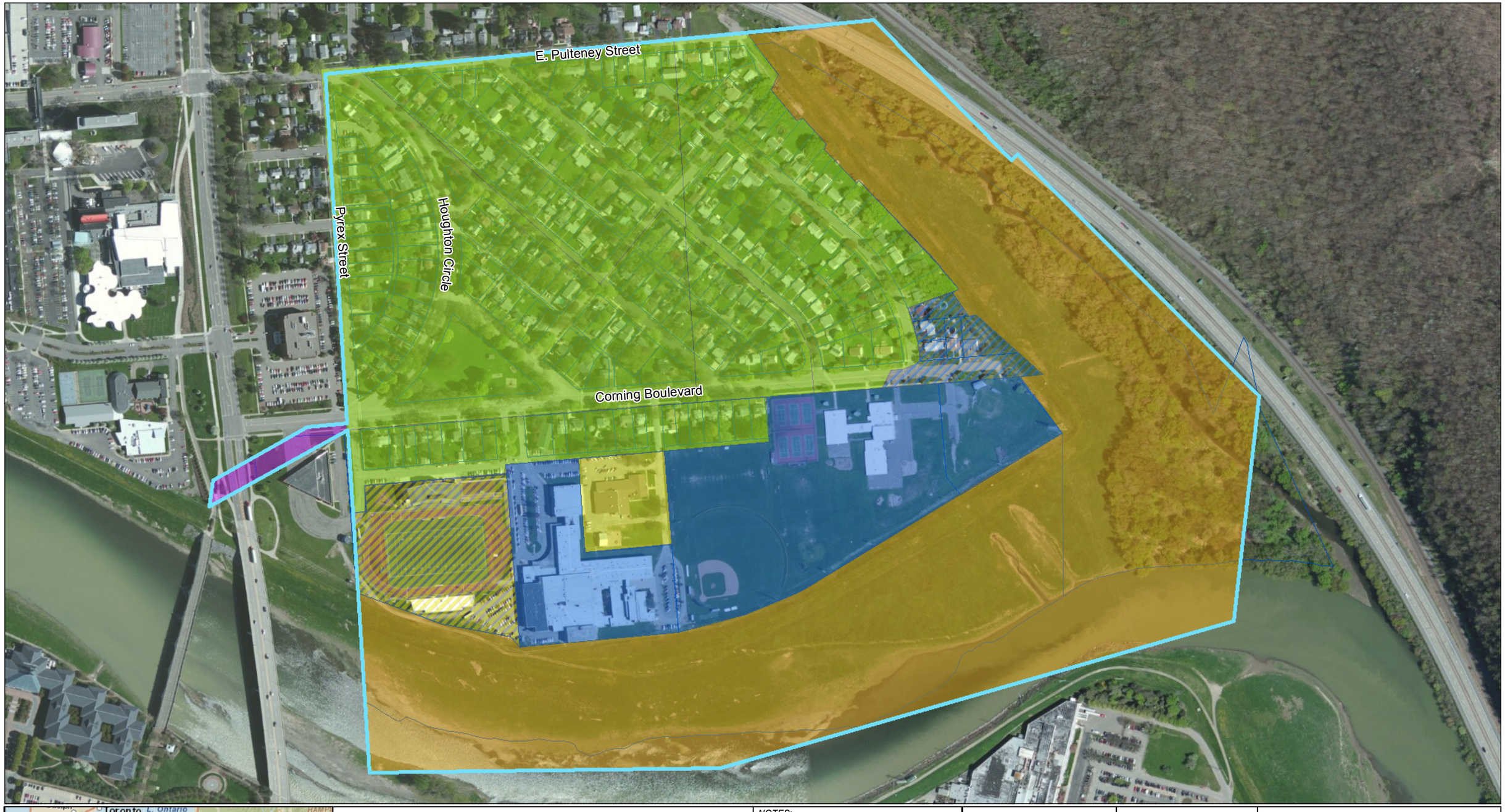
4.3.6 Flood Control Area

Initial investigations in this area will consist of field reconnaissance and visual inspection noting field conditions and indications of the presence of fill containing ash, brick and glass pieces. In particular, storm sewer outfalls that may be present in the area will be evaluated and documented. No soil sampling is anticipated in the Flood Control Area during the initial investigation, to avoid damage to the earthen dike; however, based upon initial field reconnaissance and the results of sampling activities described for other areas in the foregoing

sections, NYSDEC may request and/or Corning Incorporated may propose limited sampling in this area.

SECTION 4

FIGURES



Legend

- Study Area Boundary
- Parcels

Areas of Investigation

- Corning-Painted Post School District Property
- Corning Christian Academy
- Memorial Stadium Property
- Firehouse Frontage Area
- Residential Area at the Eastern End of Corning Blvd
- Residential Area
- Flood Control Area

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

0 350 700 Feet

0 80 160 240 Meters

WESTON SOLUTIONS

Figure 4-1
Areas of Investigation

Document Name: Areas_of_Investigation_Tabloid.MXD

6/2/2014

Legend

- Corning-Painted Post School District Property
- Parcels
- School Addition
- Former School
- Parking Lot
- Grid (100x100 ft)
- Surface Soil Sampling Locations
- Verify Existing Cover
- Soil Borings

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

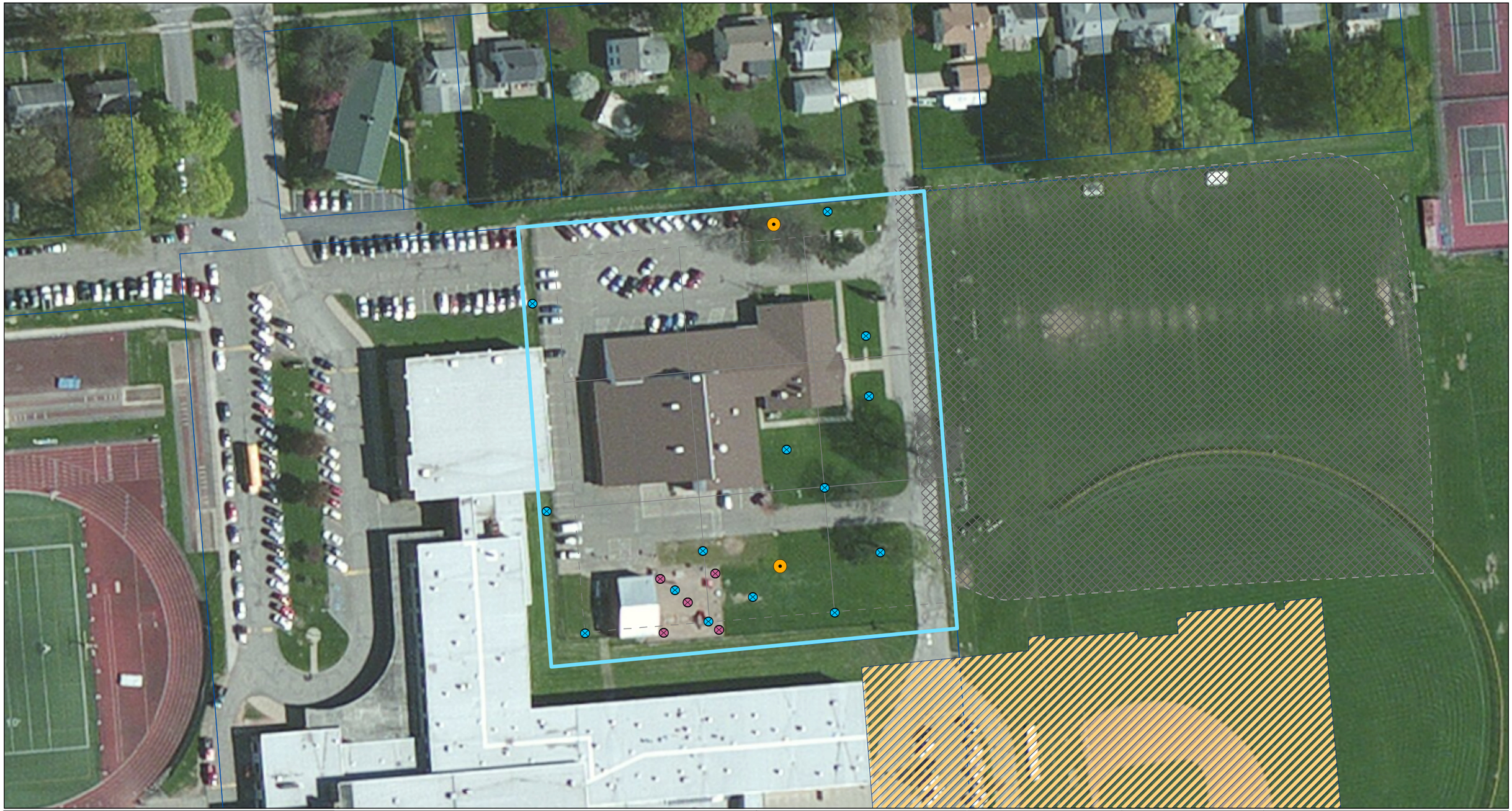
0 150 300 Feet

0 30 60 90 Meters

Figure 4-2
Characterization Activities
Corning-Painted Post
School District Property

Document Name: Proposed_Soilboring_A1.MXD

6/19/2014



Legend

- Corning Christian Academy Property
- Parcels
- Grid (100x100 ft)
- Surface Soil Sampling Locations
- Verify Existing Cover
- Soil Borings

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

0 75 150 Feet

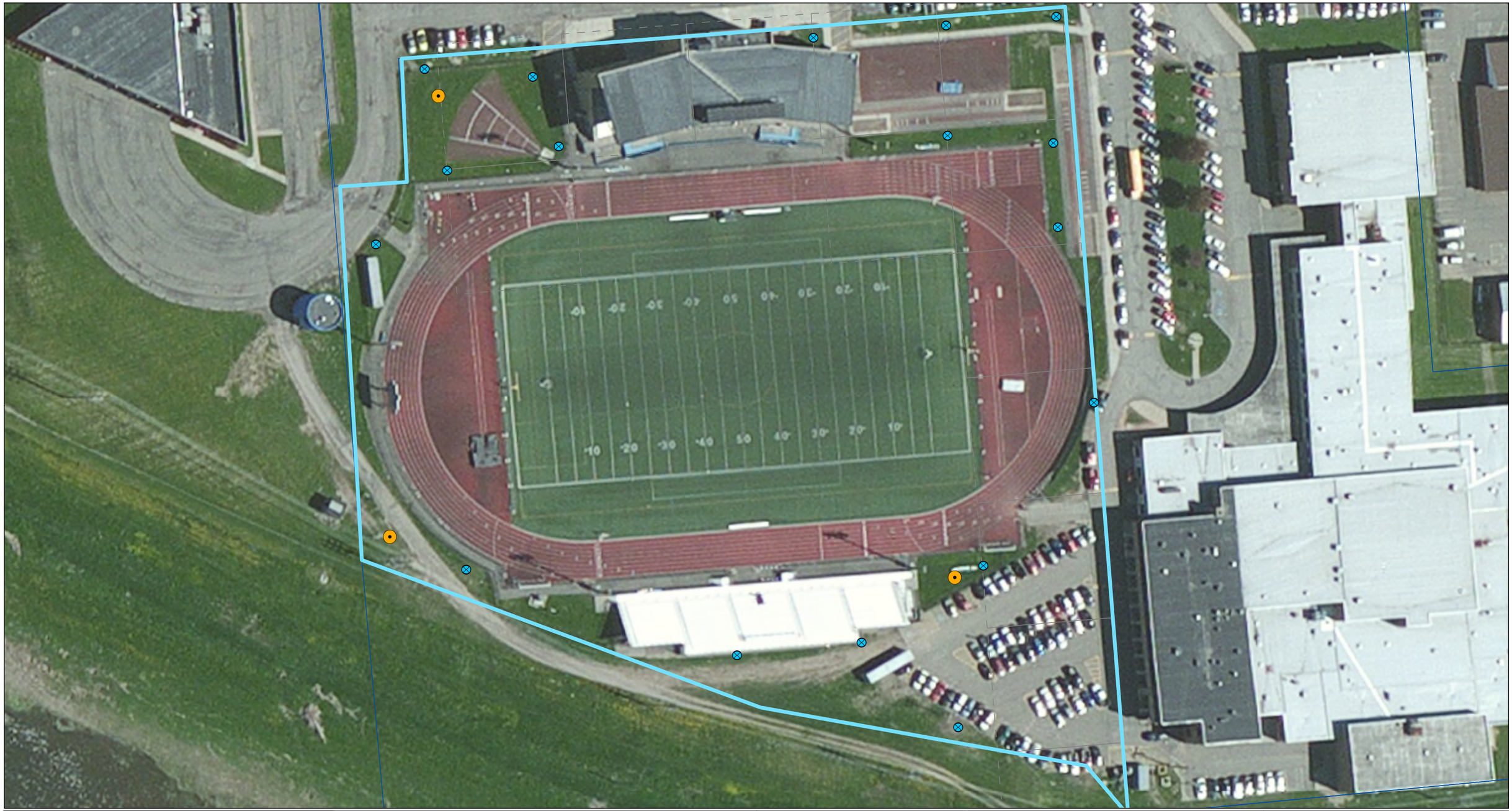
0 10 20 30 Meters

WESTON SOLUTIONS

Figure 4-3
Characterization Activities
Corning Christian Academy Property

Document Name: Proposed_Soilboring_A2.MXD

6/19/2014



Legend

- Memorial Stadium Property
- Parcels
- Grid (100x100 ft)
- Surface Soil Sampling Locations
- Soil Borings

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

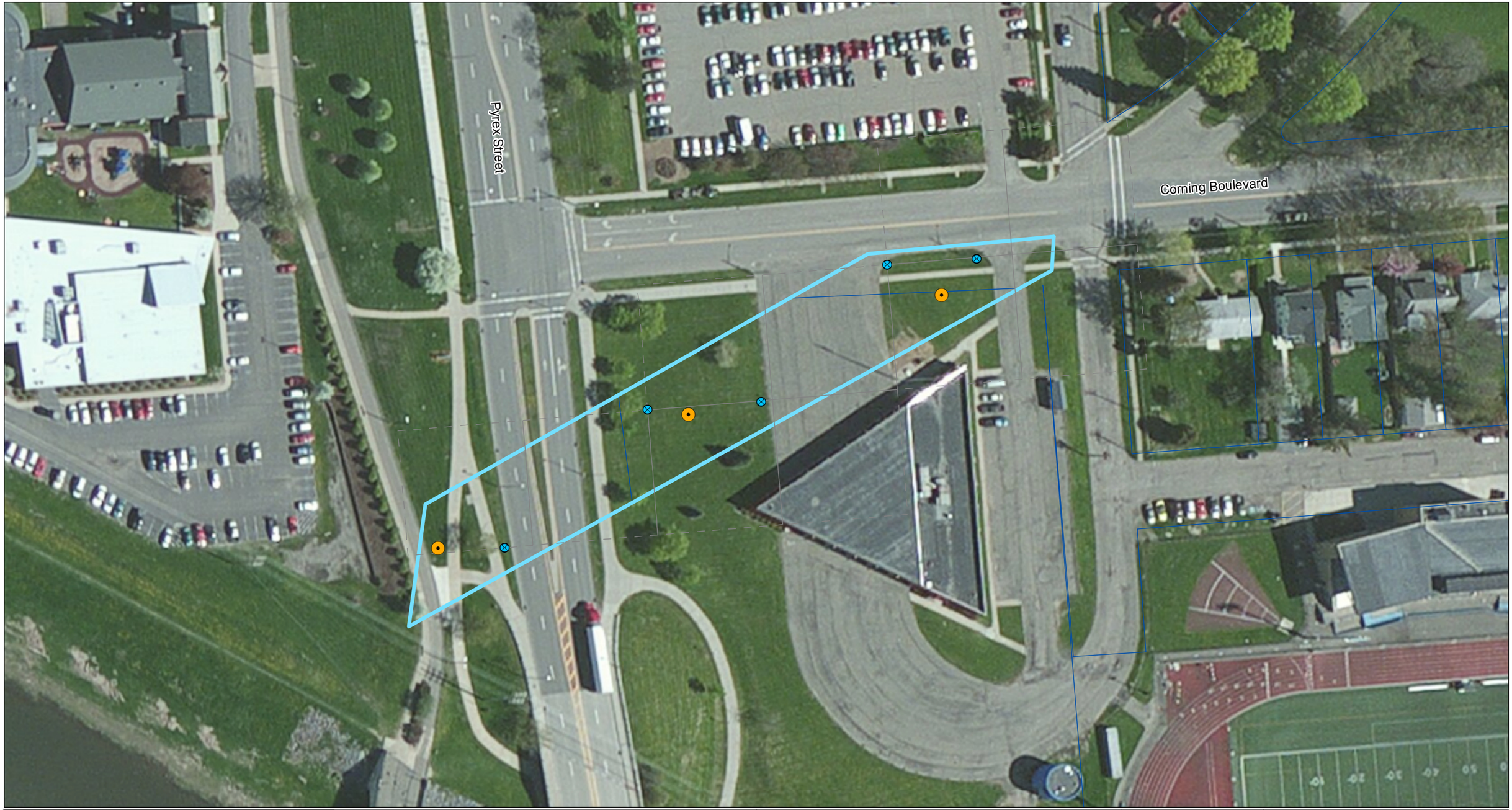
0 75 150 Feet

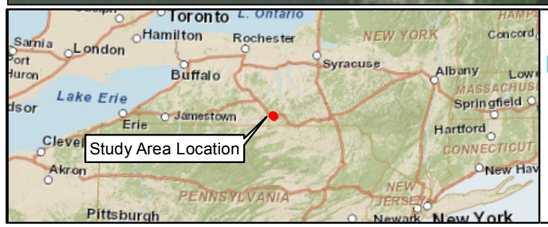
0 10 20 30 Meters

Figure 4-4
Characterization Activities
Memorial Stadium Property

Document Name: Proposed_Soilboring_A3.MXD

6/2/2014





Legend

- Firehouse Frontage Property
- Parcels
- Grid (100x100 ft)
- Soil Borings
- Surface Soil Sampling Locations

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

N

0 75 150 Feet

0 10 20 30 Meters

Study Area
Corning NY

WESTON SOLUTIONS

Figure 4-5
Characterization Activities
Firehouse Frontage Property

Document Name: Proposed_Soilboring_A4.MXD

6/2/2014

Legend

- Residential Area at the Eastern End of Corning Boulevard
- Parcels
- Area of Potential Historic Disturbance
- Soil Borings and Surface Soil Samples
- Surface Soil Sampling Locations

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

N

0 75 150 Feet

0 10 20 30 Meters

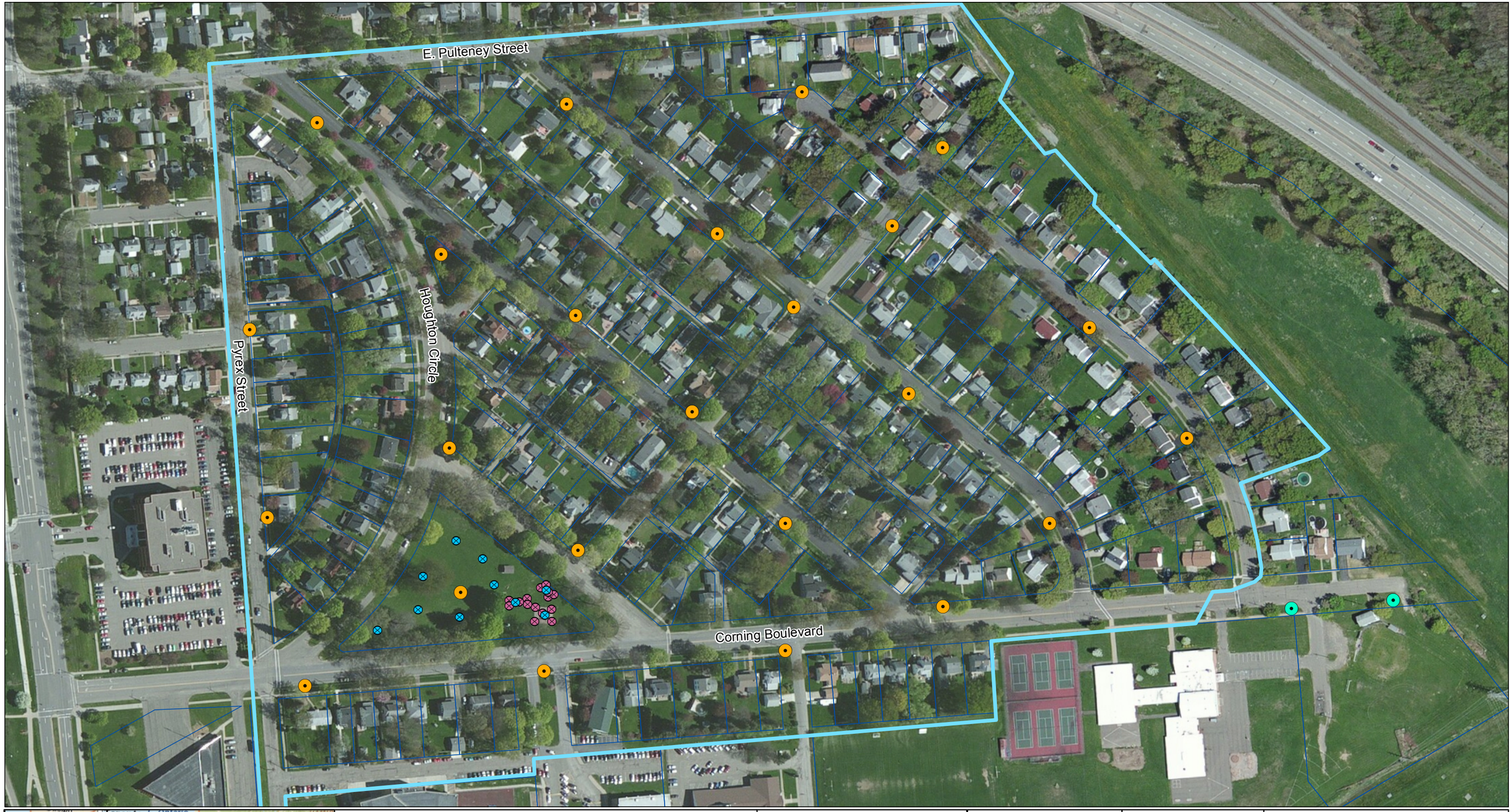
Study Area
Corning NY

WESTON SOLUTIONS

Figure 4-6
Characterization Activities
Residential Area at the
Eastern End of Corning Boulevard

Document Name: Proposed_Soilboring_A5.MXD

6/2/2014



Legend

- Residential Area
- Parcels
- Surface Soil Sampling Locations
- Verify Existing Cover
- Soil Borings
- Soil Borings in Residential Area East End of Corning Blvd

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

0 200 400 Feet

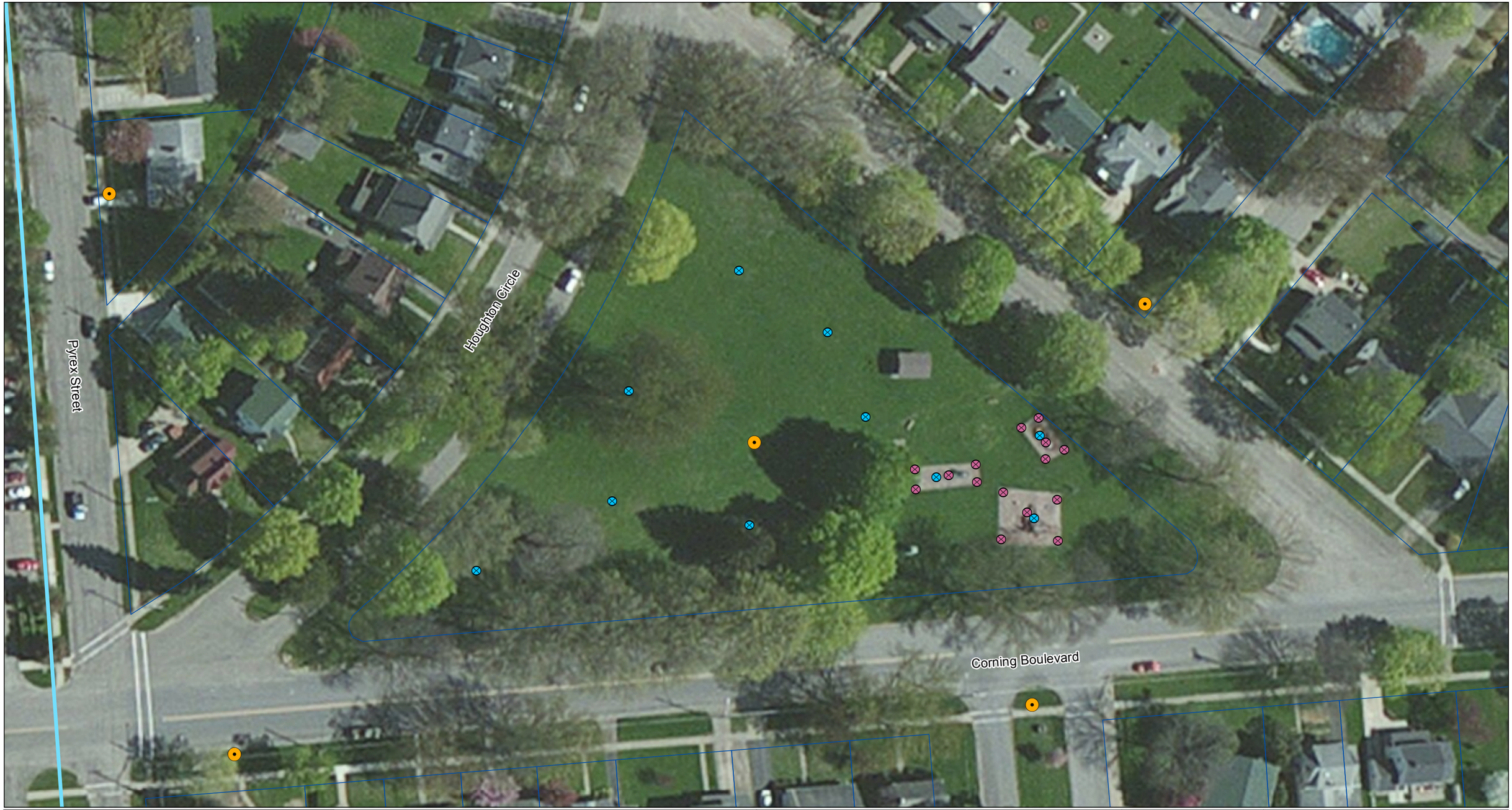
0 40 80 120 Meters

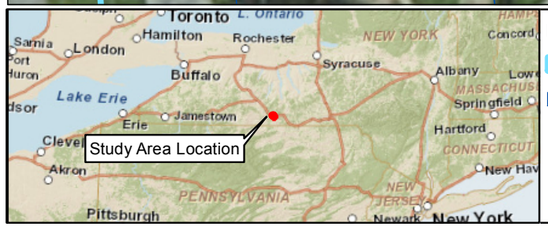
WESTON SOLUTIONS

Figure 4-7
Characterization Activities
Residential Area

Document Name: Proposed_Soilboring_A6.MXD

6/19/2014





Legend

- Residential Area
- Parcels
- Surface Soil Sampling Locations
- Verify Existing Cover
- Soil Borings

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

Study Area
Corning NY

N

0 60 120 Feet

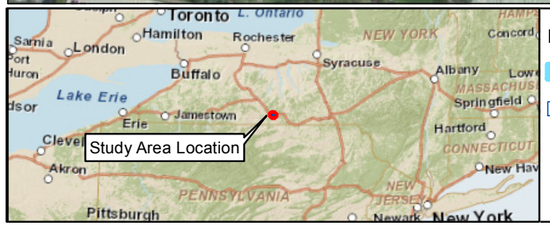
0 10 20 30 Meters

WESTON SOLUTIONS

Figure 4-8
Characterization Activities
Houghton Park in
Residential Area

Document Name: Proposed_Soilboring_Park_A6.MXD

6/2/2014



Legend

- Flood Control Area
- Parcels

Note:
Final locations and number of borings will be determined in the field.

NOTES:
Base Imagery: ESRI, DigitalGlobe, GeoEye Mapping Service, 2011
Coordinate System: NAD 1983 State Plane New York Central Feet
Datum: NAD83. Units: Feet

N

0 350 700 Feet

0 80 160 240 Meters

Study Area
Corning NY

WESTON SOLUTIONS

Figure 4-9
Flood Control Area

Document Name: Proposed_Soilboring_A8.MXD

6/2/2014

SECTION 4

TABLES

Table 4-1
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
SOIL										
Corning-Painted Post School District Property	14	Soil Borings	3	COPCs	42	3	3	0	3	51
				Metals	9	1	1	0	1	12
				TPH	9	1	1	0	1	12
				PCBs	9	1	1	0	1	12
				SVOCs	9	1	1	0	1	12
				VOCs[2]	9	1	1	1	1	13
				TCLP metals	9	1	1	0	1	12
	24	Surface Soil[3]	1	COPCs	24	2	2	0	2	30
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
	24	Shallow Soil[4]	1	COPCs	24	2	2	0	2	30
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
	50	Soil Cover	NS	NA	NS	NS	NS	NS	NS	NS
Corning Christian Academy Property	2	Soil Borings	3	COPCs	6	1	1	0	1	9
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	14	Surface Soil[3]	1	COPCs	14	1	1	0	1	17
				Metals	3	1	1	0	1	6
				TPH	3	1	1	0	1	6
				PCBs	3	1	1	0	1	6
				SVOCs	3	1	1	0	1	6
				VOCs[2]	3	1	1	1	1	7
				TCLP metals	3	1	1	0	1	6
	14	Shallow Soil[4]	1	COPCs	14	1	1	0	1	17
				Metals	3	1	1	0	1	6
				TPH	3	1	1	0	1	6
				PCBs	3	1	1	0	1	6
				SVOCs	3	1	1	0	1	6
				VOCs[2]	3	1	1	1	1	7
				TCLP metals	3	1	1	0	1	6
	5	Cover (Mulch)	NS	NA	NS	NS	NS	NS	NS	NS

Table 4-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
Memorial Stadium Property	3	Soil Borings	3	COPCs	9	1	1	0	1	12
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	17	Surface Soil[3]	1	COPCs	17	1	1	0	1	20
				Metals	4	1	1	0	1	7
				TPH	4	1	1	0	1	7
				PCBs	4	1	1	0	1	7
				SVOCs	4	1	1	0	1	7
				VOCs[2]	4	1	1	1	1	8
				TCLP metals	4	1	1	0	1	7
	17	Shallow Soil[4]	1	COPCs	17	1	1	0	1	20
				Metals	4	1	1	0	1	7
				TPH	4	1	1	0	1	7
				PCBs	4	1	1	0	1	7
				SVOCs	4	1	1	0	1	7
				VOCs[2]	4	1	1	1	1	8
				TCLP metals	4	1	1	0	1	7
Firehouse Frontage Property	3	Soil Borings	3	COPCs	9	1	1	0	1	12
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	5	Surface Soil[3]	1	COPCs	5	1	1	0	1	8
				Metals	1	1	1	0	1	4
				TPH	1	1	1	0	1	4
				PCBs	1	1	1	0	1	4
				SVOCs	1	1	1	0	1	4
				VOCs[2]	1	1	1	1	1	5
				TCLP metals	1	1	1	0	1	4
	5	Shallow Soil[4]	1	COPCs	5	1	1	0	1	8
				Metals	1	1	1	0	1	4
				TPH	1	1	1	0	1	4
				PCBs	1	1	1	0	1	4
				SVOCs	1	1	1	0	1	4
				VOCs[2]	1	1	1	1	1	5
				TCLP metals	1	1	1	0	1	4

Table 4-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
Residential Area at East End of Corning Blvd	11	Soil Borings	3	COPCs	33	2	2	0	2	39
				Metals	15	1	1	0	1	18
				TPH	15	1	1	0	1	18
				PCBs	15	1	1	0	1	18
				SVOCs	15	1	1	0	1	18
				VOCs[2]	15	1	1	1	1	19
				TCLP metals	15	1	1	0	1	18
	12	Surface Soil[3]	1	COPCs	12	1	1	0	1	15
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
Residential Area (including Houghton Park)	24	Soil Borings	3	COPCs	72	4	4	0	4	84
				Metals	15	1	1	0	1	18
				TPH	15	1	1	0	1	18
				PCBs	15	1	1	0	1	18
				SVOCs	15	1	1	0	1	18
				VOCs[2]	15	1	1	1	1	19
				TCLP metals	15	1	1	0	1	18
	866	Surface Soil[3]	1	COPCs	866	44	44	0	44	998
				Metals	214	11	11	0	11	247
				TPH	214	11	11	0	11	247
				PCBs	214	11	11	0	11	247
				SVOCs	214	11	11	0	11	247
				VOCs[2]	214	11	11	1	11	248
				TCLP metals	214	11	11	0	11	247
	15	Cover (Mulch)	NS	NA	NS	NS	NS	NS	NS	NS
									TOTAL SOIL ANALYSES:	**3,594**

Table 4-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
GROUNDWATER										
School Area	5	Monitoring Wells	2	COPCs	10	2	0	0	2	14
				Metals	2	2	0	0	2	6
				TPH	2	2	0	0	2	6
				PAH	2	2	0	0	2	6
				PCBs	2	2	0	0	2	6
				VOCs	2	2	0	2	2	8
Residential Area at East End of Corning Blvd	4	Monitoring Wells	2	COPCs	8	2	0	0	2	12
				Metals	2	2	0	0	2	6
				TPH	2	2	0	0	2	6
				PAH	2	2	0	0	2	6
				PCBs	2	2	0	0	2	6
				VOCs	2	2	0	2	2	8
TOTAL GROUNDWATER ANALYSES:										**90**

Notes:

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

[2] - VOCs collected at highest concentration(s) in borings where photoionization detector readings are >5X background

[3] - Surface samples collected from 0 - 2 inches bgs

[4] - Shallow samples collected from 2 inches bgs to 2 feet bgs

No samples collected of soil cover - confirmation of existence and thickness only

TPH - Total Petroleum Hydrocarbons

PAH - Polycyclic aromatic hydrocarbons

PCB - Polychlorinated biphenyls

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

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 analyzed for list (as shown)

All samples analyzed for COPCs, 20% of samples analyzed for full list

DUP, FB and MS/MSD collected at 20%

TB is 1 per VOC cooler (assume 1 per area)

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
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 lidd	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)]
Groundwater				
TAL Metals	SW846846 6010	250 mL, Polyethlyene or Glass	$HN0_3$ to pH < 2	6 months
TPH	EPA 1664 (SGT HEM)	1000 mL, Glass with Teflon®-lined cap	4°C, H_2S0_4 or HCl to pH < 2	28 days
PAH	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
PAH - Polycyclic aromatic hydrocarbons
PCB - Polychlorinated biphenyls
VOCs - 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

Table 4-3
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	0.00200	0.000300
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
Total Petroleum Hydrocarbons (TPH) [Method EPA 1664 (SGT HEM)]	**mg/Kg**		**mg/L**	
TPH	100	40.0	5.00	1.94
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270]	**µg/Kg**		**µg/L**	
Biphenyl	170	10.5	5.00	0.653
bis (2-chloroisopropyl) ether	170	17.6	5.00	0.520
2,4,5-Trichlorophenol	170	36.8	5.00	0.480
2,4,6-Trichlorophenol	170	11.1	5.00	0.610
2,4-Dichlorophenol	170	8.85	5.00	0.510
2,4-Dimethylphenol	170	45.6	5.00	0.500
2,4-Dinitrophenol	330	59.1	10.0	2.22
2,4-Dinitrotoluene	170	26.1	5.00	0.447
2,6-Dinitrotoluene	170	41.3	5.00	0.400
2-Chloronaphthalene	170	11.3	5.00	0.460
2-Chlorophenol	170	8.59	5.00	0.530
2-Methylphenol	170	5.19	5.00	0.400
2-Methylnaphthalene	170	2.04	5.00	0.600
2-Nitroaniline	330	54.1	10.0	0.420
2-Nitrophenol	170	7.72	5.00	0.480
3,3'-Dichlorobenzidine	170	148	5.00	0.400
3-Nitroaniline	330	38.8	10.0	0.480
4,6-Dinitro-2-methylphenol	330	58.3	10.0	2.20
4-Bromophenyl phenyl ether	170	53.7	5.00	0.450
4-Chloro-3-methylphenol	170	6.94	5.00	0.450
4-Chloroaniline	170	49.5	5.00	0.590
4-Chlorophenyl phenyl ether	170	3.60	5.00	0.350
4-Methylphenol	330	9.40	10.0	0.360
4-Nitroaniline	330	18.9	10.0	0.250
4-Nitrophenol	330	40.9	10.0	1.52
Acenaphthene	170	1.98	5.00	0.410
Acenaphthylene	170	1.38	5.00	0.380
Acetophenone	170	8.66	5.00	0.540
Anthracene	170	4.32	5.00	0.280
Atrazine	170	7.51	5.00	0.460
Benzaldehyde	170	18.5	5.00	0.267
Benzo[a]anthracene	170	2.91	5.00	0.360
Benzo[a]pyrene	170	4.07	5.00	0.470
Benzo[b]fluoranthene	170	3.28	5.00	0.340
Benzo[g,h,i]perylene	170	2.03	5.00	0.350
Benzo[k]fluoranthene	170	1.86	5.00	0.730
Bis(2-chloroethoxy)methane	170	9.18	5.00	0.350

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

	Soil		Groundwater	
	RL	MDL	RL	MDL
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270] (continued)	µg/Kg		µg/L	
Bis(2-chloroethyl)ether	170	14.6	5.00	0.400
Bis(2-ethylhexyl) phthalate	170	54.4	5.00	1.80
Butyl benzyl phthalate	170	45.3	5.00	0.420
Caprolactam	170	73.0	5.00	2.20
Carbazole	170	1.95	5.00	0.300
Chrysene	170	1.69	5.00	0.330
Dibenz(a,h)anthracene	170	1.99	5.00	0.420
Di-n-butyl phthalate	170	58.3	5.00	0.310
Di-n-octyl phthalate	170	3.95	5.00	0.470
Dibenzofuran	170	1.76	10.0	0.510
Diethyl phthalate	170	5.10	5.00	0.220
Dimethyl phthalate	170	4.40	5.00	0.360
Fluoranthene	170	2.45	5.00	0.400
Fluorene	170	3.89	5.00	0.360
Hexachlorobenzene	170	8.39	5.00	0.510
Hexachlorobutadiene	170	8.64	5.00	0.680
Hexachlorocyclopentadiene	170	51.0	5.00	0.590
Hexachloroethane	170	13.1	5.00	0.590
Indeno[1,2,3-cd]pyrene	170	4.67	5.00	0.470
Isophorone	170	8.44	5.00	0.430
N-Nitrosodi-n-propylamine	170	13.4	5.00	0.540
N-Nitrosodiphenylamine	170	9.23	5.00	0.510
Naphthalene	170	2.81	5.00	0.760
Nitrobenzene	170	7.48	5.00	0.290
Pentachlorophenol	330	57.9	10.0	2.20
Phenanthrene	170	3.54	5.00	0.440
Phenol	170	17.8	5.00	0.390
Pyrene	170	1.09	5.00	0.340
2-Fluorobiphenyl				
Polychlorinated Biphenyls (PCBs) [Method SW846 8082]	mg/Kg		ug/L	
PCB-1016	0.0167	0.00326	0.500	0.176
PCB-1221	0.0167	0.00326	0.500	0.176
PCB-1232	0.0167	0.00326	0.500	0.176
PCB-1242	0.0167	0.00326	0.500	0.176
PCB-1248	0.0167	0.00326	0.500	0.176
PCB-1254	0.0167	0.00782	0.500	0.250
PCB-1260	0.0167	0.00782	0.500	0.250
PCB-1262	0.0167	0.00782	0.500	0.250
PCB-1268	0.0167	0.00782	0.500	0.250
Volatile Organic Compounds (VOCs) [Method SW846 8260]	ug/Kg		ug/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-Trichloroethane	5.00	0.650	1.00	0.230
1,1,2-Trichloro-1,2,2-trifluoroethane	5.00	1.14	1.00	0.310
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-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
Bromoform	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
Dibromochloromethane	5.00	0.640	1.00	0.320
Chloroethane	5.00	1.13	1.00	0.320
Chloroform	5.00	0.309	1.00	0.340

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

	Soil		Groundwater	
	RL	**MDL**	**RL**	**MDL**
Volatile Organic Compounds (VOCs) [Method SW846 8260] (continued)	**ug/Kg**		**ug/L**	
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
Dichlorodifluoromethane	5.00	0.413	1.00	0.680
Ethylbenzene	5.00	0.345	1.00	0.740
1,2-Dibromoethane	5.00	0.642	1.00	0.730
Isopropylbenzene	5.00	0.754	1.00	0.790
Methyl acetate	5.00	0.930	2.50	0.500
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
TCLP Metals [Method SW846 6010]	**mg/L**		**--**	
Aluminum	0.200	0.0600	--	--
Antimony	0.0200	0.00679	--	--
Arsenic	0.0150	0.00555	--	--
Barium	0.00200	0.000700	--	--
Beryllium	0.00200	0.000300	--	--
Boron	0.0200	0.00400	--	--
Cadmium	0.00200	0.000500	--	--
Calcium	0.500	0.100	--	--
Chromium	0.00400	0.00100	--	--
Cobalt	0.00400	0.000630	--	--
Copper	0.0100	0.00160	--	--
Iron	0.0500	0.0193	--	--
Lead	0.0100	0.00300	--	--
Magnesium	0.200	0.0434	--	--
Manganese	0.00300	0.000400	--	--
Nickel	0.0100	0.00126	--	--
Potassium	0.500	0.100	--	--
Selenium	0.0250	0.00870	--	--
Silver	0.00600	0.00170	--	--
Sodium	1.00	0.324	--	--
Thallium	0.0200	0.0102	--	--
Vanadium	0.00500	0.00150	--	--
Zinc	0.0100	0.00150	--	--

Notes:
mg/Kg - milligram per kilogram
ug/Kg - microgram per kilogram
mg/L- milligram per liter
ug/L - microgram per liter

5. PROJECT MANAGEMENT

5.1 SCHEDULE

The activities described in this 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 characterization activities will occur in a phased approach and the anticipated project schedule is provided as Figure 5-1. It should be noted that the planned activities are not independent and the proposed schedule and approach may be adjusted accordingly as work is completed within the Study Area. The schedule will be updated bi-weekly 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 contain a variety of 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 characterization activities to provide photo documentation of field activities. In particular, photos of the soil boring cores will be collected 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. NYSDEC and NYSDOH will be provided verbal updates of the field activities periodically and electronic copies of weekly work activity reports, including select supporting photographs. 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 NYSDOH on scheduled basis (i.e. daily for levels which require actions, weekly for routine monitoring data).

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. All data obtained during the characterization activities will be summarized in a Study Area Characterization Report and the associated laboratory analytical data packages will be included as an attachment.

Laboratory data deliverable packages will be reviewed for completeness, adherence to holding times, comparison with chain-of-custody, etc. Data validation will be performed, and a data usability summary report (DUSR) will be prepared. The data review/validation activities are described in the QAPP provided in Appendix C.

5.2.5 Reporting

Following implementation of characterization investigation activities, a Study Area Characterization Report documenting the investigation activities and findings will be prepared and submitted to NYSDEC. This report will include a summary of the activities, including a description of any deviations from the proposed work plan, as well as the submission of analytical results including the results of the QA/QC samples.

The aforementioned report will typically contain the following information:

- Tables summarizing sample analytical results. Final laboratory data packages will be appended to this report.
- Summary table presenting depth to water measurements and water level elevations at the Study Area monitoring wells.
- Maps showing soil and groundwater sampling locations.
- Water level elevation contour map prepared using data collected from the monitoring wells.
- Stratigraphic boring logs and monitor well construction summaries.
- Discussion of the sampling results and significance of findings.

Upon receipt of validated data as it is generated throughout the project, Corning Incorporated will supply such validated data to NYSDEC. NYSDEC will review the data and Corning Incorporated's proposed letters before they are provided to individual property owners.

5.3 HEALTH AND SAFETY PLAN

The health and safety of field workers, clients, 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 Study Area Health and Safety Plan (HASP) and Community Air Monitoring Plan (CAMP) provided in Appendix A and Appendix B, respectively.

5.4 STUDY AREA CONTROLS

The first activities will involve mobilization of personnel and equipment. A temporary field office and equipment staging area will be set up near the Study Area. This temporary field office area will be surrounded by temporary fencing for security. The office area and access gate will be closed and locked when not in use. The location of the temporary field office and equipment storage area will be determined based on the written access consent agreements.

The temporary field office area will consists of an office trailer for document and sample preparation, and staging area for field equipment. Electricity will be supplied to the mobile office via either a power drop or a generator.

5.5 COMMUNITY RELATIONS

A 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 Work Plan and other Study Area related activities.

SECTION 5

FIGURES

CHARACTERIZATION WORK PLAN PROPOSED SCHEDULE[1]

TASK	EVENT	WEEKS 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56
Initial Startup	Consent Order																																																										
	Public Meeting																																																										
	Mobilization																																																										
Corning-Painted Post School District Property	Obtain Written Access Consent & Clearances																																																										
	Geophysical Survey																																																										
	Soil Boring Sampling																																																										
	Soil Boring Sample Analysis & Validation																																																									
	Data Report for School District property (Soil Boring / Geophysics)[2]																																																									
	Surface Soil (measurement & sampling)																																																									
	Surface Soil Sample Analysis & Validation																																																									
	Data Report for School District property (Surface Soil)[2]																																																									
	Potential Groundwater Installation, Sampling and Reporting																																																									
Corning Christian Academy	Obtain Written Access Consent & Clearances																																																										
	Soil Boring & Surface Soil Sampling																																																									
	Sample Analysis & Validation																																																									
	Data Report for Corning Christian Academy property[2]																																																									
Memorial Stadium & Firehouse Frontage	Obtain Written Access Consent & Clearances																																																									
	Soil Boring & Surface Soil Sampling																																																									
	Sample Analysis & Validation																																																									
	Data Report for City of Corning[2]																																																									
Residential Area at the East End of Corning Blvd	Obtain Written Access Consent & Clearances																																																									
	Soil Boring & Surface Soil Sampling																																																									
	Sample Analysis & Validation																																																									
	Data reports to Property Owners[2]																																																									
	Potential Groundwater Installation, Sampling and Reporting																																																									
Residential Area	Obtain Written Access Consent (City of Corning) & Clearances																																																									
	Soil Boring Sampling (Right-of-Way Areas)																																																									
	ROW Soil Boring Sample Analysis & Validation																																																									
	Data Report to City of Corning[2]																																																									
	Obtain Written Access Consent (Property Owners - Phase I) & Clearances																																																									
	Surface Soil Sampling (Phase I)																																																									
	Surface Soil Sample Analysis & Validation																																																									
	Data Report for Property Owners[2]																																																									
	Obtain Written Access Consent (Property Owners - Phase II) & Clearances																																																									
	Surface Soil Sampling (Phase II)																																																									
	Surface Soil Sample Analysis & Validation																																																									
	Data Report for Property Owners[2]																																																									
Flood Control Area	Obtain Written Access Consent																																																									
	Field Reconnaissance																																																									
Summary Report	Prepare Draft Characterization Report																																																									
	NYSDEC Comments																																																									
	Prepare Final Characterization Report																																																									

Notes:

1 -Schedule is predicated upon obtaining written access consent from property owners

2 - Assumed 2 weeks for NYSDCE review of submittals.

Figure 5-1
Project Schedule

6. REFERENCES

Aerial photograph of the Corning, New York area, August 8, 1938

Aerial photograph of the Corning, New York area, May 8, 1942

Aerial photograph of the Corning, New York area, April 16, 1952

Aerial photograph of the Corning, New York area, July 11, 1955

Aerial photograph of the Corning, New York area, October 8, 1964

Aerial photograph of the Corning, New York area, March 30, 1968

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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.

HEALTH AND SAFETY PLAN (*HASP*)

Office:	West Chester, PA
Project Name:	Study Area Bounded by Pyrex Street, E. Pulteney Street, Post Creek and Chemung River
Client:	Corning Incorporated
Work Location:	Corning, NY
WO#:	02005.056.001.0001

HEALTH AND SAFETY PLAN (*HASP*)

Work Order Number	Date	Project Manager Approval	Project Safety Manager Approval

HEALTH AND SAFETY PLAN (HASP)

Prepared by: A. Jayne / R. McLoughlin	**W.O. Number:** 02005.056.001.0001	**Date:** 03/28/2014

Project Identification: Study Area Bounded by Pyrex St., E. Pulteney St., Post Creek and Chemung River

Office: West Chester, PA

Site Name: Study Area, Corning, New York

Client: Corning Incorporated

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

History:
Soil and/or groundwater characterization activities at a site with potential fill containing ash, brick and glass pieces.

Scope of Work: Study Area Characterization Activities

☐ **S**ite visit only; site HASP not necessary. List personnel here and sign off below:

X Utility notification required. If required, provide utility notification agency, authorization number, and valid dates:

Regulatory Status:

Site regulatory status: TBD

CERCLA/SARA	**RCRA**	**Other Federal Agency**
☐ U.S. EPA	☐ U.S. EPA	☐ DOE
☐ State	☐ State	☐ USACE
☐ NPL Site	**NRC**	☐ Air Force
☐ OSHA	☐ 10 CFR 20	☐ _____

Hazard Communication (Req'd See Attachment D)
☐ 1910 ☐ 1926 ☐ State

Safety Officer Manual (Required to be On-Site)
Based on the Hazard Assessment and Regulatory Status, determine the Standard HASP(s) applicable to this project. Indicate below which Standard HASP will be used and append the appropriate pages of this form along with the Standard Plan.

☐ Stack Test	☐ _____
☐ Air Emissions	☐ _____
☐ Asbestos	☐ _____
☐ Industrial Hygiene	☐ _____
☐ _____	☐ _____

Review and Approval Documentation:

Reviewed by: SO/DEHSM/CEHS	George Crawford (Name Print)	(Signature)	Date:
Environmental. Compliance Advisor	(Name Print)	(Signature)	Date:
Approved by: Project Manager	John Sontag (Name Print)	(Signature)	Date:

Hazard Assessment and Equipment Selection:

In accordance with WESTON's Personal Protective Equipment Program and 29 CFR 1910.132, at the site prior to personnel beginning work, the FSO and/or the Site Manager have evaluated conditions and verified that the personal protective equipment selection outlined within this HASP is appropriate for the hazards known or expected to exist. (Refer to CEHS Program Manual Section 5, Personal Protection Program, for guidance.)

☒ **FSO**	Dave Cairns (Name)	(Signature)	Date:
☒ **Site Manager**	John Sontag (Name)	(Signature)	Date:

☒ **Project Environmental Compliance Officer**	John Sontag (Name)	Date:
☒ **Dangerous Goods Shipping Coordinator**	Rachel McLoughlin (Name)	Date:

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.

_____ _____

_____ _____

TABLE OF CONTENTS

Section **Page**

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

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/Project Scientist	1400 Weston Way West Chester, PA 19380	610-701-3428
National Accounts	Dave Cairns/ Senior Geoscientist	1400 Weston Way West Chester, PA 19380	610-701-3676

Roles and Responsibilities:

Manage and implement site characterization program.

1.2 WESTON SUBCONTRACTORS

Organization/Branch	Name/Title	Address	Telephone
	Name: Title:	Street: City: State, Zip:	
	Name: Title:	Street: City: State, Zip:	
	Name: Title:	Street: City: State, Zip:	

Roles and Responsibilities:

SITE-SPECIFIC HEALTH AND SAFETY PERSONNEL

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

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; current Adult First Aid and CPR certification; familiarity with jobs of similar scope.

Designated alternates include: John Sontag, Rachel McLoughlin

WESTON SOLUTIONS

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: Dave Cairns** **Title: Senior Geoscientist** **Task(s): All** **Certification Level or Description:** ☒ Medical Current ☒Training Current ☐Fit Test Current (Qual.) ☐Fit Test Current (Quant.)
Name: Rachel McLoughlin **Title: Project Scientist** **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.)
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.)

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: TBD
Address:

Activities To Be Conducted by Subcontractor:

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, which has been accepted by Project PM.

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

June 2014

2.1 HEALTH AND SAFETY EVALUATION

2.1.1 Task Hazard Assessment

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

Activities Covered Under This Plan:

No.	Task/Subtask	Description	Schedule
1	Soil sampling	A combination of soil boring and surface sampling.	2014
2	Groundwater investigation	Installation of groundwater monitoring wells and groundwater sampling	2014

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
- ☐ O_2 Rich
- ☐ O_2 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:

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

Comments:

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 ( )	Chemical Name	Quantity
Arsenic			
Lead			
Cadmium			

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	

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 (µCii/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 - Benzene Exposure Control Plan
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

3. SITE SECURITY

3.1 SITE SECURITY ASSESSMENT FORM

DESCRIPTION	
Site Name and Location: Former Study Area, Corning NY	**Number of Employees and Subcontractors on Site:** TBD
Type of Work: Study Area characterization sampling activities (Soil and/or groundwater sampling)	
Projected Start Date: 2014	**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 *(urban/suburban/rural; residential/commercial/industrial; traffic volume, population density, etc*	
Suburban, residential neighborhood with school property within Study Area limits.	
THREAT INDICATORS *(apparent social, economic, political, ethnic, criminal, gang related, and other risk factors)*	
N/A	
COUNTERMEASURES *(Current and projected risk mitigation factors)*	
Security Systems (Reference Site Security Checklist):	
Security Procedures (Reference Site Security Checklist):	
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 *(Submit "Medium" and "High" risk assessments to Corporate Security for review)*	
Risk Level: ☒ **Low** ☐ **Medium** ☐ **High**	**Date:**
Site Safety Officer:	**Division Safety Manager:**
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)	☐ / ☐	☐ / ☐
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		
b. Site Supervisor		
c. Project Manager	John Sontag	610-701-3679
d. PC Manager		

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

4. TASK BY TASK ASSESSMENT

4.1 TASK-BY-TASK RISK ASSESSMENT

4.1.1 Task 1 Description

TASK 1: Soil sampling. Includes a combination of soil boring and surface soil sampling.

EQUIPMENT REQUIRED/USED

Geoprobe and/or Hollow-stem auger rig
Scoops
Nitrile gloves
Safety Boots
Safety Glasses
Dust Monitoring
Hand tools
Hearing Protection
Mini Rae

POTENTIAL HAZARDS/RISKS

Chemical

☒ Hazard Present Risk Level: ☐ H ☐ M ☒ L
What justifies risk level?
Sampling soil with potential metals.

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 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?

LEVELS OF PROTECTION/JUSTIFICATION

Level D

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.
FLD 02, 05, 06, 10,11, 12, 13, 20, 22, 28, 34, 37, 38, 41, 43, 47, 56, 57, 59, 60, Section 7.0, Environmental Remediation Drilling Safety Guidance – 2005.

TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.2 Task 2 Description

TASK 2: Groundwater sampling activities, includes the installation of groundwater monitoring wells and groundwater sampling

EQUIPMENT REQUIRED/USED

Hollow-stem auger Rig	**Hand Tools**	**Dust Monitoring**
Nitrile Gloves	**Sample Bottles**	
Safety Boots	**Water Level Indicator**	
Safety Glasses	**Groundwater Pumps**	
Hearing Protection	**Bailers**	
MiniRae	**Tubing**	

POTENTIAL HAZARDS/RISKS

Chemical

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

What justifies risk level?

Ground water sampling with potential constituents at lower levels

Physical

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

What justifies risk level?

Work generally will occur at residential or school property, with some work possibly in 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?

LEVELS OF PROTECTION/JUSTIFICATION

Level D

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.

FLD 01, 02, 05, 06, 10, 11, 12, 17, 18, 19, 20, 32, 34, 35, 36, 37, 41, 43, 47, 57, 59, 60 Section 7.0, Environmental Remediation Drilling Safety Guidance – 2005.

4.1 TASK-BY-TASK RISK ASSESSMENT (Continued)

4.1.3 Task 3 Description

EQUIPMENT REQUIRED/USED

POTENTIAL HAZARDS/RISKS

Chemical

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

Physical

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

Biological

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

RADIOLOGICAL

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

LEVELS OF PROTECTION/JUSTIFICATION

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.

4.2 PERSONNEL PROTECTION PLAN

Engineering Controls
Describe Engineering Controls used as part of Personnel Protection Plan:

Task(s)	
Tasks 1-2	

Administrative Controls
Describe Administrative Controls used as part of Personnel Protection Plan:

Task(s)	Tasks 1-2
All	Conduct hazard analysis of all work tasks.
All	Conduct safety briefings with contractors prior to performing daily tasks to discuss safety hazards and controls Taken to minimize or eliminate hazards

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)	Tasks 1-2
All	Hard hat, safety glasses, safety shoes, hearing protection (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	☐ Head
☒ Eye and Face	Safety Glasses	☐ Eye and Face
☒ Hearing	Ear plugs in designated areas	☐ 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	**Task(s):** NA
☐ Head	☐ Head
☐ Eye and Face	☐ Eye and Face
☐ Hearing	☐ Hearing
☐ Arms and Legs Only	☐ Arms and Legs Only
☐ Whole Body	☐ Whole Body
☐ Apron	☐ Apron
☐ Hand – Gloves	☐ Hand - Gloves
☐ Gloves	☐ Gloves
☐ Gloves	☐ Gloves
☐ Foot - Safety Boots	☐ Foot - Safety Boots
☐ Outer Boots	☐ Outer Boots
☐ Boots (Other)	☐ Boots (Other)
☐ Half Face	☐ SAR - Airline
☐ Cart./Canister	☐ SCBA
☐ Full Face	☐ Comb. Airline/SCBA
☐ Cart./Canister	☐ Cascade System
☐ PAPR	☐ Compressor
☐ Cart./Canister	☐ Fall Protection
☐ Type C	☐ Flotation
☐ Fall Protection	☐ Other
☐ 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			☐		
☒ MiniRAE				☐		
☐ MultiRAE (LEL/O2/H2S/CO/PID)				☐		
☐ TVA 1000 (PID/FID)				☐		
☐ Other _____				☐		
☐ **FID**						
☐ TVA 1000 (FID/PID)				☐		
☐ Other _____	1, 2			☐		
☒ **PDR 1000 (Particulate)**				☐		
☐ **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

Instrument, Mfg., Model, Equip. ID No.	Date	Time	Calib. Material	Calib. Method Mfg.'s	Other	Initial Setting and Reading	Final Setting and Reading	Calibrator's Initials

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		Continue work.
		3 times background to < 1 mR/hour		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, 2	1.0 units sustained		Increase monitoring frequency. Stop work and evaluate appropriate PPE
☒ **Inorganic Gases, Vapors, and Particulates**	1, 2	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.

5.3 ACTION LEVELS

(Attach action level calculations)

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	James Davis	(251) 434-6420 - (334) 319-0380 (cell)
WESTON Medical Programs Manager	William Irwin	(610) 701-3684 - (267) 918-8371 (cell)
WESTON Health & Safety Division Safety Manager	George Crawford	(610) 701-3771 - (484) 437-5976 (Cell)
WESTON Health & Safety Local Safety Officer	George Crawford	(610) 701-3771- (484) 437-5976 (Cell)
Fire Department		911
Police Department		911
WESTON FSO Cell Phone		
WESTON PM Cell Phone	John Sontag	(610) 701-3679
Client Site Phone		
Site Telephone		
Nearest Telephone		
Poison Control		(800) 222-1222

Local Medical Emergency Facility(s) - LMF

Name of Hospital: Guthrie Corning Hospital		
Address: 176 Denison Pkwy E, Corning, NY 14830		Phone No.: 607-937-7200
Name of Contact:		Phone No.:
Type of Service: **X** Physical trauma only ☐ Chemical exposure only ☐ Physical trauma and chemical exposure ☐ Available 24 hours	**Route to Hospital:** **(See Attached)**	**Travel time from site:** 4 Minutes **Distance to hospital:** 0.8 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:** **/**

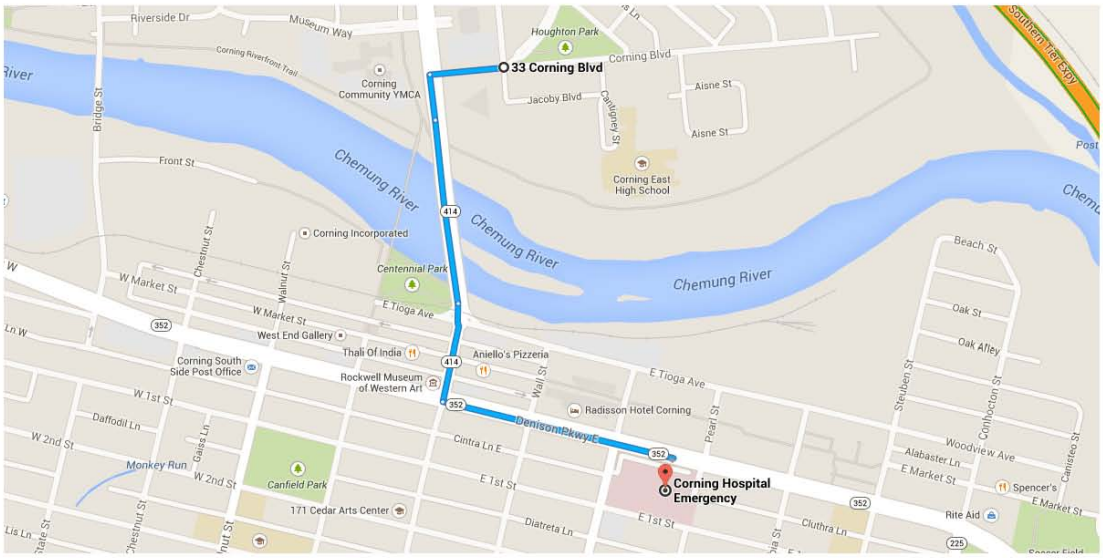
See reporting an incident in Attachment F.

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.

Google

Directions from **33 Corning Blvd** to **Corning Hospital Emergency** **Drive 0.8 mi, 4 min**



33 Corning Blvd
Corning, NY 14830

1. Head **west** on **Corning Blvd** toward **Center Way** — 472 ft
2. Turn **left** onto **Center Way** — 292 ft
3. Continue onto **Brisco Bridge** — 0.2 mi
4. **Brisco Bridge** turns slightly **right** and becomes **Cedar St** — 0.1 mi
5. Turn **left** onto **Denison Pkwy E** — 0.3 mi
   Destination will be on the right

Corning Hospital Emergency
176 Denison Pkwy E, Corning, NY 14830

6.1 CONTINGENCIES

6.1.3 Response Plans

<table>
<tr><td rowspan="3">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</td><td>First Aid Kit:
☒ Yes ☐ No

Blood Borne Pathogens Kit:
☒ Yes ☐ No</td><td>Type
Appropriate sized ANSI-approved Type III Kit, plus BBP</td><td>Location
In Vehicle near work area</td><td>Special First-Aid Procedures:
Cyanides on-site
☐ Yes ☒ No
If yes, contact LMF. Do they have antidote kit?
☐ Yes ☐ No</td></tr>
<tr><td>Eyewash required
☒ Yes ☐ No</td><td>Type</td><td>Location</td><td>HF on-site
☐ Yes ☒ No
If yes, need neutralizing ointment for first-aid kit. Contact LMF.</td></tr>
<tr><td>Shower required
☐ Yes ☒ No</td><td>Type</td><td>Location</td><td></td></tr>
</table>

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.	a. Cleanup per SDSs if small; or sound alarm, call for assistance, notify Emergency Coordinator 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 only if safe and trained in its use e. Stand by to inform emergency responders of materials and conditions	Type/Location ABC/Vehicle / / / / / /
Description of Spill Response Gear	Location	Description (Other Fire Response Equipment)		Location

Plan to Respond to Security Problems 911 Emergency

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

Provide a description of waste disposition including identification of storage area, hauler, and final disposal site, if applicable

Drill cuttings and other waste soil/water generated during characterization activities will be containerized daily. The filled containers will be staged in a secure, designated area. The waste soil and waste water will be properly disposed.

Equipment Decontamination

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

Equipment will be decontaminated in accordance with the decontamination procedures described in the Work Plan.

Sampling Equipment Decontamination

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

All non-dedicated sampling and monitoring equipment will be decontaminated in accordance with the decontamination procedures described in the Work Plan.

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	
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:

Disposal Plan, End of Week:

Disposal Plan, End of Project:

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:

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:

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 l	☐ 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	☐

8.2 HEALTH AND SAFETY PLAN APPROVAL/SIGNOFF FORM

Site Name: Study Area, Corning, New York	**WO#:** 02005.056.001.0001
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

ATTACHMENT A
CHEMICAL CONTAMINANTS DATA SHEETS

June 2014

ATTACHMENT B
SAFETY DATA SHEETS

(ATTACH SDS)

June 2014

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

June 2014

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: TBD

☐ List of chemicals compiled, format: **x** HASP ☐ Other: _____

☐ Location of SDS files: Attached

☐ Training conducted by: Name: TBD Date: _____

☐ Indicate format of training documentation: **X** 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 **X** 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.

Chemicals in Unlabeled Pipes

Work activities may be performed by employees in areas where chemicals are transferred through unlabeled pipes. Prior to starting work in these areas, the employee will contact the SO, at which time information as to the chemical(s) in the pipes, potential hazards of the chemicals or the process involved, and the safety precautions that should be taken will be determined and presented.

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

June 2014

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**

AIR MONITORING/SAMPLING DATA LOG

Client:	**W.O. No.:**	**Sample No.:**
Address:	**Sampled By:**	**Date:**

Employee and Location Information

Employee Name:	**Employee No.:**	**Job Title**:

Respirator ☐ APR ☐ ½ Mask ☐ Full Face ☐ PAPR ☐ ½ Mask ☐ Full Face ☐ Hood ☐ SAR ☐ ½ Mask ☐ Full Face ☐ Hood ☐ SCBA	**Manufacturer:**	**Cartridge Type:**

PPE: ☐ Hard Hat ☐ HPD ☐ Gloves ☐ Safety Shoes ☐ Coveralls ☐ Other:

Sampling Data

Sampling Type: ☐ Personal ☐ TWA ☐ STEL ☐ Area ☐ Source ☐ Full Shift ☐ Partial Shift ☐ Grab	**Media:**	**Pump Type/Serial No.:** /
Calibrator/Serial No.: /	**Pre-Calibration:** 1. 2. 3. **avg-pre:**	**Post-Calibration:** 1. 2. 3. **avg-post**:

Start Time:	**Restart Time:**	**Restart Time:**	**Avg. Flow rate:**	**% Change:**
1st Stop Time:	**2nd Stop Time:**	**3rd Stop Time:**	**Total Time:**	**Volume:**

Multiple Samples for this TWA: ☐ Yes ☐ No	**Multiple Chemical Exposures:** ☐ Yes ☐ No	**Exposure Time:** ☐ Normal ☐ Worst Case

Sampling Conditions

Weather Conditions: Temp: R.H: B.P.: Other:

Engineering Controls:

Substances Evaluated

Substance	Result	Substance	Result	Substance	Result

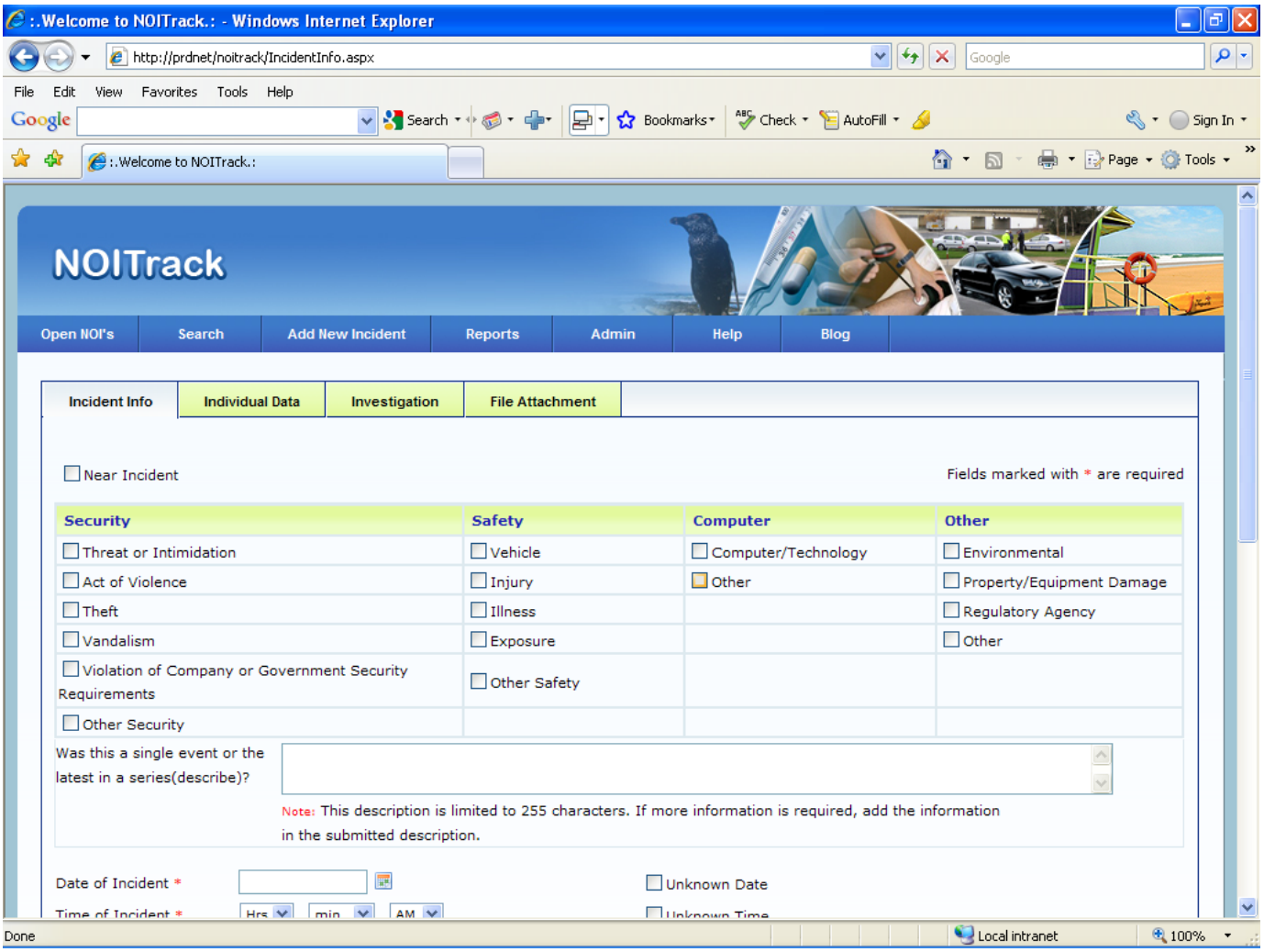
Observations and Comments

QA by: _____ **Date:** _____

June 2014

ATTACHMENT F
INCIDENT REPORTING

June 2014



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.

June 2014

ATTACHMENT G
TRAFFIC CONTROL PLAN

June 2014

ATTACHMENT H
ENVIRONMENTAL HEALTH & SAFETY INSPECTION CHECKLIST

June 2014

ENVIRONMENTAL HEALTH AND SAFETY INSPECTION CHECKLIST

Project Name: _____

Inspector: _____

Submit to: _____

Date: _____

June 2014

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?	

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?	

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?	

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, 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, 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, slivers 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 failing 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 -/= to four vertical to twelve horizontal and with edges 6 ft or more above 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 Bounded by Pyrex Street, E. Pulteney Street, Post Creek and Chemung River
Corning, NY
NYSDEC Project IS 851046

June 2014

Prepared for

Corning Incorporated
Corning, New York

Prepared by

WESTON SOLUTIONS, INC.
West Chester, Pennsylvania 19380

W.O. No. 02005.056.001.0001

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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
$\mu g/m^3$	micrograms per cubic meter
NYSDEC	New York State Department of Environmental Conservation
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 characterization activities at the Study Area located in Corning, New York, bounded by Pyrex Street on the west, E. Pulteney Street on the north, Post Creek on the east and the Chemung River on the south (Study Area). This air monitoring plan will supplement the existing Health and Safety Plan (HASP) and provide an additional measure of protection to potential receptors not directly involved with the characterization activities.

As presented in the Study Area Characterization Work Plan (Work Plan), intrusive characterization activities planned to be conducted within the Study Area may include subsurface soil sampling and groundwater investigations. Since the primary constituents of potential concern (COPCs) at the Study Area are arsenic, cadmium, and lead, air monitoring for dust particulates and dust control techniques will be performed during intrusive activities to provide an additional measure of protection to the surrounding community.

2. METHODS

Perimeter air monitoring for dust particles will be conducted at a minimum of two stations, one generally located upwind, and one generally located downwind of any intrusive characterization activity. In addition, due to the close proximity of playgrounds, athletic playing fields, schools and childcare centers, 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. 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 characterization activities will change, the location of the monitoring point relative to the activity will be modified as needed and documented. 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 intrusive activities such as 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 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 $\mu g/m^3$ 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 $\mu g/m^3$ greater than background (measured at the upwind location). If the downwind particulate levels exceed 150 $\mu g/m^3$ 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 $\mu g/m^3$ 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 Bounded by Pyrex Street, E. Pulteney Street,
Post Creek and Chemung River
Corning, NY
NYSDEC Project ID 851046

June 2014

Prepared for

Corning Incorporated
Corning, New York

Prepared by

WESTON SOLUTIONS, INC.
West Chester, Pennsylvania 19380

W.O. No. 02005.056.001.

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ATTACHMENT A	TestAmerica Quality Assurance Manual and Standard Operating Procedures

LIST OF ACRONYMS

ASP	Analytical Services Protocol
ASTM	American Society of Testing and Materials
BS	blank spike
BSD	blank spike duplicate
CAMP	Community Health and Safety Plan
cfs	cubic feet per second
COPC	constituents of potential concern
CPR	cardiopulmonary resuscitation
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
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
OSHA	Occupational Safety and Health Administrations
PID	Photoionization Detector
PPE	personal protective equipment
QA	quality assurance
QAM	TestAmerica Quality Assurance Manual
QAO	quality assurance objectives
QAPP	Quality Assurance Project Plan
QC	quality control

LIST OF ACRONYMS (CONTINUED)

RPD	relative percent difference
SCO	site cleanup objectives
SOP	standard operating procedure
SOW	scope of work
USEPA	U.S. Environmental Protection Agency
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 at the Study Area bounded by Pyrex Street on the west, E. Pulteney Street on the north, Post Creek on the east and the Chemung River on the south (Study Area) in Corning, New York.

1.1 PROJECT SCOPE AND GOALS

The purpose of the characterization activities detailed in the Study Area Characterization Work Plan (Work Plan) is to assess the potential presence and nature of fill material within the Study Area. Accordingly, Corning Incorporated is conducting a historic records search and review to establish a history of the Study Area and identify areas where fill material may potentially have been placed. This records search includes a review of historic aerial photographs to identify areas where historic disturbances may have occurred. The Work Plan includes a summary of the historic records review and a plan for characterization activities based on the preliminary results of the historic records review. The characterization activities described in the Work Plan are designed to assess whether potential fill material is present within the Study Area and to develop data necessary for understanding the current conditions within the Study Area and associated potential exposure pathways.

The specific objectives of the Work Plan are as follows:

1. In areas where historic records indicate potential disturbances:
    a. assess the nature and extent of the potential disturbance area, and
    b. assess potential exposure pathways, in the event fill material is found.
2. In areas where historic records do not indicate potential disturbances, evaluate the potential presence of fill material.

Additional details, including figures of proposed sampling locations are included in the Work Plan.

1.2 PROJECT DATA QUALITY AND OBJECTIVES

This QAPP documents the QA/QC measures that will be followed during the implementation of 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 the Study Area.

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;
- Well abandonment;
- 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 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 Work Plan are of known and acceptable level of quality.

Primary DQOs will include the following:

- Complete the Study Area characterization activities to adequately confirm the presence/absence of fill materials;
- Complete the Study Area characterization activities to adequately confirm the presence/absence of constituents at concentrations greater than reasonable quantitation limits; and
- Provided 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 be 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 the analytical results will be to generate sufficient information to determine the presence or absence of Constituents of Potential Concern (COPCs) within the Study Area's media and to determine the presence/absence of and/or the nature and extent of COPCs at the Study Area. The project data manager will track data from collection of samples through login at the laboratory to delivery by technical report and electronic data delivery, oversee necessary validation/data usability summary report preparation (DUSR), and coordinate laboratory corrective actions.

The following sections discuss the steps to be taken to ensure the quality of data acquired during the work. The representativeness of the measurement data is a function of the sampling strategy and will be achieved by following the procedures in the 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 the TestAmerica's QA Manual (QAM) found in Attachment A.

1.4 PROJECT SCHEDULE

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

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 a 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 Study Area. Mr. Ford is responsible for primary contact with New York State Department of Environmental Control (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
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HP-ME-03-83
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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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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 and Mr. John Sontag will lead the Field Team and serve as Health and Safety Officer and be responsible for oversight of environmental 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 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. Marta Cairns of WESTON will be responsible for managing the analytical data generated from the project activities.

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marta.cairns@westonsolutions.com

2.3 LABORATORY STAFFING

Analytical work required during the project activities will be performed by TestAmerica Laboratories, Inc. (TestAmerica), located in Buffalo, NY. TestAmerica is a NYSDEC Environmental Laboratory Approval Program (ELAP)-certified laboratory (certification #10026). 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 QAM is included as Attachment A of this QAPP. The laboratory QAM includes a detailed explanation 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 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

Brian Fisher 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.

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.

Brian Fisher – Project Manager
TestAmerica Buffalo
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716-504-9800

Brad Prinzi – Laboratory Quality Manager
TestAmerica Buffalo
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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, including medical monitoring, Occupational Safety and Health Administrations (OSHA) - related training, first aid/cardiopulmonary resuscitation (CPR), equipment operation, and associated records and documentation, can be found in the HASP prepared for the sampling activity. 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 number of samples for characterization activities are presented in the sample summary table (see Table 3-1). Sampling locations, rationale, and analytical methods, as well as the sampling and decontamination procedures, for this project are discussed in detail in the Work Plan and attached SOPs.

Prior to the Study Area characterization 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 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 Work Plan). In accordance with the 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 Work Plan. As described in Section 4, final selection of drilling locations will depend on securing all necessary clearances, 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 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 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 Work Plan (see Section 4).

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

- Locations will be cleared by an underground utility survey (as needed).
- Soil cuttings 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 3-2. Additional information regarding the analytical methods is specified in the TestAmerica QAM.

3.4 GROUNDWATER SAMPLING PROCEDURES

The Work Plan includes a groundwater investigation program, which will be developed during the characterization activities and contingent upon soil characterization results. As described in the Work Plan, the groundwater investigation program will likely involve the installation of groundwater monitoring wells and potential sampling of existing wells (dependent upon access agreements with the well owners). Additional details regarding groundwater investigation approach is included in the Work Plan (See Section 4).

The groundwater sampling procedure, as described in the Work Plan, contains of 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 QAM.

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 Table 3-1.

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 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 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 3-3.

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 Laboratory will follow SOPs (see Attachment A of this QAPP) for handling, identification, control, and chain-of-custody procedures and to maintain the validity of the samples. The laboratory custody procedures are presented in the TestAmerica QAM (Attachment A of this QAPP).

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
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
SOIL										
Corning-Painted Post School District Property	14	Soil Borings	3	COPCs	42	3	3	0	3	51
				Metals	9	1	1	0	1	12
				TPH	9	1	1	0	1	12
				PCBs	9	1	1	0	1	12
				SVOCs	9	1	1	0	1	12
				VOCs[2]	9	1	1	1	1	13
				TCLP metals	9	1	1	0	1	12
	24	Surface Soil[3]	1	COPCs	24	2	2	0	2	30
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
	24	Shallow Soil[4]	1	COPCs	24	2	2	0	2	30
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
	50	Soil Cover	NS	NA	NS	NS	NS	NS	NS	NS
Corning Christian Academy Property	2	Soil Borings	3	COPCs	6	1	1	0	1	9
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	14	Surface Soil[3]	1	COPCs	14	1	1	0	1	17
				Metals	3	1	1	0	1	6
				TPH	3	1	1	0	1	6
				PCBs	3	1	1	0	1	6
				SVOCs	3	1	1	0	1	6
				VOCs[2]	3	1	1	1	1	7
				TCLP metals	3	1	1	0	1	6
	14	Shallow Soil[4]	1	COPCs	14	1	1	0	1	17
				Metals	3	1	1	0	1	6
				TPH	3	1	1	0	1	6
				PCBs	3	1	1	0	1	6
				SVOCs	3	1	1	0	1	6
				VOCs[2]	3	1	1	1	1	7
				TCLP metals	3	1	1	0	1	6
	5	Cover (Mulch)	NS	NA	NS	NS	NS	NS	NS	NS

Table 3-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
Memorial Stadium Property	3	Soil Borings	3	COPCs	9	1	1	0	1	12
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	17	Surface Soil[3]	1	COPCs	17	1	1	0	1	20
				Metals	4	1	1	0	1	7
				TPH	4	1	1	0	1	7
				PCBs	4	1	1	0	1	7
				SVOCs	4	1	1	0	1	7
				VOCs[2]	4	1	1	1	1	8
				TCLP metals	4	1	1	0	1	7
	17	Shallow Soil[4]	1	COPCs	17	1	1	0	1	20
				Metals	4	1	1	0	1	7
				TPH	4	1	1	0	1	7
				PCBs	4	1	1	0	1	7
				SVOCs	4	1	1	0	1	7
				VOCs[2]	4	1	1	1	1	8
				TCLP metals	4	1	1	0	1	7
Firehouse Frontage Property	3	Soil Borings	3	COPCs	9	1	1	0	1	12
				Metals	2	1	1	0	1	5
				TPH	2	1	1	0	1	5
				PCBs	2	1	1	0	1	5
				SVOCs	2	1	1	0	1	5
				VOCs[2]	2	1	1	1	1	6
				TCLP metals	2	1	1	0	1	5
	5	Surface Soil[3]	1	COPCs	5	1	1	0	1	8
				Metals	1	1	1	0	1	4
				TPH	1	1	1	0	1	4
				PCBs	1	1	1	0	1	4
				SVOCs	1	1	1	0	1	4
				VOCs[2]	1	1	1	1	1	5
				TCLP metals	1	1	1	0	1	4
	5	Shallow Soil[4]	1	COPCs	5	1	1	0	1	8
				Metals	1	1	1	0	1	4
				TPH	1	1	1	0	1	4
				PCBs	1	1	1	0	1	4
				SVOCs	1	1	1	0	1	4
				VOCs[2]	1	1	1	1	1	5
				TCLP metals	1	1	1	0	1	4

Table 3-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
Residential Area at East End of Corning Blvd	11	Soil Borings	3	COPCs	33	2	2	0	2	39
				Metals	15	1	1	0	1	18
				TPH	15	1	1	0	1	18
				PCBs	15	1	1	0	1	18
				SVOCs	15	1	1	0	1	18
				VOCs[2]	15	1	1	1	1	19
				TCLP metals	15	1	1	0	1	18
	12	Surface Soil[3]	1	COPCs	12	1	1	0	1	15
				Metals	5	1	1	0	1	8
				TPH	5	1	1	0	1	8
				PCBs	5	1	1	0	1	8
				SVOCs	5	1	1	0	1	8
				VOCs[2]	5	1	1	1	1	9
				TCLP metals	5	1	1	0	1	8
Residential Area (including Houghton Park)	24	Soil Borings	3	COPCs	72	4	4	0	4	84
				Metals	15	1	1	0	1	18
				TPH	15	1	1	0	1	18
				PCBs	15	1	1	0	1	18
				SVOCs	15	1	1	0	1	18
				VOCs[2]	15	1	1	1	1	19
				TCLP metals	15	1	1	0	1	18
	866	Surface Soil[3]	1	COPCs	866	44	44	0	44	998
				Metals	214	11	11	0	11	247
				TPH	214	11	11	0	11	247
				PCBs	214	11	11	0	11	247
				SVOCs	214	11	11	0	11	247
				VOCs[2]	214	11	11	1	11	248
				TCLP metals	214	11	11	0	11	247
	15	Cover (Mulch)	NS	NA	NS	NS	NS	NS	NS	NS
									TOTAL SOIL ANALYSES:	**3,594**

Table 3-1 (continued)
Sample Summary Table

Area	No. Sample Locations		Estimated No. Samples per Location	Analysis[1]	No. Primary Samples	Estimated No. QC Samples				Total
						DUP	FB	TB	MS/MSD	
GROUNDWATER										
School Area	5	Monitoring Wells	2	COPCs	10	2	0	0	2	14
				Metals	2	2	0	0	2	6
				TPH	2	2	0	0	2	6
				PAH	2	2	0	0	2	6
				PCBs	2	2	0	0	2	6
				VOCs	2	2	0	2	2	8
Residential Area at East End of Corning Blvd	4	Monitoring Wells	2	COPCs	8	2	0	0	2	12
				Metals	2	2	0	0	2	6
				TPH	2	2	0	0	2	6
				PAH	2	2	0	0	2	6
				PCBs	2	2	0	0	2	6
				VOCs	2	2	0	2	2	8
TOTAL GROUNDWATER ANALYSES:										**90**

Notes:

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

[2] - VOCs collected at highest concentration(s) in borings where photoionization detector readings are >5X background

[3] - Surface samples collected from 0 - 2 inches bgs

[4] - Shallow samples collected from 2 inches bgs to 2 feet bgs

No samples collected of soil cover - confirmation of existence and thickness only

TPH - Total Petroleum Hydrocarbons

PAH - Polycyclic aromatic hydrocarbons

PCB - Polychlorinated biphenyls

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

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 analyzed for list (as shown)

All samples analyzed for COPCs, 20% of samples analyzed for full list

DUP, FB and MS/MSD collected at 20%

TB is 1 per VOC cooler (assume 1 per area)

Table 3-2
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
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 lidd	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)
Groundwater				
TAL Metals	SW846846 6010	250 mL, Polyethlyene or Glass	$HN0_3$ to pH < 2	6 months
TPH	EPA 1664 (SGT HEM)	1000 mL, Glass with Teflon®-lined cap	4°C, H_2S0_4 or HCl to pH < 2	28 days
PAH	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
PAH - Polycyclic aromatic hydrocarbons
PCB - Polychlorinated biphenyls
VOCs - 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

Table 3-3
Field Sample Identifiers

Soil Samples						
Site Location	**Sample Type**	**Sample Area**	**Sample Number**	**QC Type**	**Depth/Location/Date**	**Date**
CONY - Corning, NY	SS- Soil Sample	CPP = Corning-Painted Post School District Property	SS### - Soil Sample #	0 - Primary Sample	#### - Depth at Top of Sample	YYMMDD = Year/Month/Date
	SB-Soil Boring	CCA = Corning Christian Academy	SB### - Soil Boring #	FB- Field Blank Sample	(e.g. 2.0 ft is 0020)	
		ROW = Right-of-Way Area		MS-Matrix Spike		
		CMS = Memorial Stadium Area		MSD-Matrix Spike Duplicate		
		FFR = Firehouse Frontage		DUP - Duplicate Sample		
		RES = Residential Area				
		FCA = Flood Control Area				

Example ID: CONY-SB-EECSB05-0-0002-140601

Primary soil sample collected from soil boring #5 in the Eastern End of Corning Blvd Area collected at 0.2 ft bgs on June 1, 2014

Water Samples					
Site Location	**Sample Type**	**Sample Area**	**Sample Location**	**QC Type**	**Date**
CONY - Corning, NY	GW - Groundwater Sample	CPP = Corning-Painted Post School District Property	MW## - Temporary Well Sample Point	0 - Primary Sample	YYMMDD = Year/Month/Date
		CCA = Corning Christian Academy	TRIP## -Trip Blank Sample Point	FB - Field Blank Sample	
		ROW = Right-of-Way Area	FIELD## - Field Blank Sample Point	TB - Trip Blank Sample	
		CMS = Memorial Stadium Area		MS - Matrix Spike	
		FFR = Firehouse Frontage		MSD - Matrix Spike Duplicate	
		RES = Residential Area		DUP - Duplicate Sample	
		FCA = Flood Control Area			

Example ID: CONY-GW-CPPMW01-0-140601

Primary groundwater sample collected from monitoring well #1 in the Corning-Painted Post School District Area on June 1, 2014

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Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc.

Client Contact	**Project Manager:**	**Site Contact:**	**Date:**	COC No:
Your Company Name here	**Tel/Fax:**	**Lab Contact:**	**Carrier:**	_____ of _____ COCs
Address	**Analysis Turnaround Time**			Sampler:
City/State/Zip	☐ CALENDAR DAYS ☐ WORKING DAYS			**For Lab Use Only:**
(xxx) xxx-xxxx Phone	TAT if different from Below _____			Walk-in Client:
(xxx) xxx-xxxx FAX	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)															Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for_____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ Therm ID No.:_____

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.2, dated 04/02/2013

FIGURE 3-1 TestAmerica Chain-of-Custody

4. FIELD OPERATIONS

This section includes brief descriptions of field procedures used to conduct environmental evaluations at the Study Area. 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 include the following:

- Soil sampling and borehole log forms;
- Field logbooks;
- 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.

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. Designated field sheets may also be used to record project activities. 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 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 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 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 eVor 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
- PM_{10} 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 PM_{10} 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;
- 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 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) will perform analysis on environmental samples where certification exists. The laboratory will follow QA/QC procedures specified by the analytical methods. Analytical results will meet the method detection limits (MDLs) 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). Table 5-1 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 3-2 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 III, December 1996 (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 compounds/analytes 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 Manual 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 its QAM (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 electronic data delivery (EDD) for all samples with QC sample data to be utilized during the data review/validation 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 blank spike and blank spike duplicate (BS/BSD) data as a measure of analytical accuracy and as a measure of analytical precision, where applicable. BS/BSD data will be compared to laboratory acceptance criteria for the maximum RPD.

- Review of laboratory control sample (LCS) data as a measure of analytical accuracy, where applicable. LCS 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 validation will be performed, and a DUSR will be prepared in accordance with DER-10 Appendix 2B. This DUSR will be prepared by a scientist capable of conducting a full data validation. The DUSR will provide the assessment included in the initial data review discussed above, with further related QA/QC information consideration, enabling full evaluation of the analytical data's usability and quality.

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

Table 5-1
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	0.00200	0.000300
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
Total Petroleum Hydrocarbons (TPH) [Method EPA 1664 (SGT HEM)]	**mg/Kg**		**mg/L**	
TPH	100	40.0	5.00	1.94
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270]	**µg/Kg**		**µg/L**	
Biphenyl	170	10.5	5.00	0.653
bis (2-chloroisopropyl) ether	170	17.6	5.00	0.520
2,4,5-Trichlorophenol	170	36.8	5.00	0.480
2,4,6-Trichlorophenol	170	11.1	5.00	0.610
2,4-Dichlorophenol	170	8.85	5.00	0.510
2,4-Dimethylphenol	170	45.6	5.00	0.500
2,4-Dinitrophenol	330	59.1	10.0	2.22
2,4-Dinitrotoluene	170	26.1	5.00	0.447
2,6-Dinitrotoluene	170	41.3	5.00	0.400
2-Chloronaphthalene	170	11.3	5.00	0.460
2-Chlorophenol	170	8.59	5.00	0.530
2-Methylphenol	170	5.19	5.00	0.400
2-Methylnaphthalene	170	2.04	5.00	0.600
2-Nitroaniline	330	54.1	10.0	0.420
2-Nitrophenol	170	7.72	5.00	0.480
3,3'-Dichlorobenzidine	170	148	5.00	0.400
3-Nitroaniline	330	38.8	10.0	0.480
4,6-Dinitro-2-methylphenol	330	58.3	10.0	2.20
4-Bromophenyl phenyl ether	170	53.7	5.00	0.450
4-Chloro-3-methylphenol	170	6.94	5.00	0.450
4-Chloroaniline	170	49.5	5.00	0.590
4-Chlorophenyl phenyl ether	170	3.60	5.00	0.350
4-Methylphenol	330	9.40	10.0	0.360
4-Nitroaniline	330	18.9	10.0	0.250
4-Nitrophenol	330	40.9	10.0	1.52
Acenaphthene	170	1.98	5.00	0.410
Acenaphthylene	170	1.38	5.00	0.380
Acetophenone	170	8.66	5.00	0.540
Anthracene	170	4.32	5.00	0.280
Atrazine	170	7.51	5.00	0.460
Benzaldehyde	170	18.5	5.00	0.267
Benzo[a]anthracene	170	2.91	5.00	0.360
Benzo[a]pyrene	170	4.07	5.00	0.470
Benzo[b]fluoranthene	170	3.28	5.00	0.340
Benzo[g,h,i]perylene	170	2.03	5.00	0.350
Benzo[k]fluoranthene	170	1.86	5.00	0.730
Bis(2-chloroethoxy)methane	170	9.18	5.00	0.350

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

	Soil		Groundwater	
	RL	MDL	RL	MDL
Semi-Volatile Organic Compounds (SVOCs) [Method SW846 8270] (continued)	µg/Kg		µg/L	
Bis(2-chloroethyl)ether	170	14.6	5.00	0.400
Bis(2-ethylhexyl) phthalate	170	54.4	5.00	1.80
Butyl benzyl phthalate	170	45.3	5.00	0.420
Caprolactam	170	73.0	5.00	2.20
Carbazole	170	1.95	5.00	0.300
Chrysene	170	1.69	5.00	0.330
Dibenz(a,h)anthracene	170	1.99	5.00	0.420
Di-n-butyl phthalate	170	58.3	5.00	0.310
Di-n-octyl phthalate	170	3.95	5.00	0.470
Dibenzofuran	170	1.76	10.0	0.510
Diethyl phthalate	170	5.10	5.00	0.220
Dimethyl phthalate	170	4.40	5.00	0.360
Fluoranthene	170	2.45	5.00	0.400
Fluorene	170	3.89	5.00	0.360
Hexachlorobenzene	170	8.39	5.00	0.510
Hexachlorobutadiene	170	8.64	5.00	0.680
Hexachlorocyclopentadiene	170	51.0	5.00	0.590
Hexachloroethane	170	13.1	5.00	0.590
Indeno[1,2,3-cd]pyrene	170	4.67	5.00	0.470
Isophorone	170	8.44	5.00	0.430
N-Nitrosodi-n-propylamine	170	13.4	5.00	0.540
N-Nitrosodiphenylamine	170	9.23	5.00	0.510
Naphthalene	170	2.81	5.00	0.760
Nitrobenzene	170	7.48	5.00	0.290
Pentachlorophenol	330	57.9	10.0	2.20
Phenanthrene	170	3.54	5.00	0.440
Phenol	170	17.8	5.00	0.390
Pyrene	170	1.09	5.00	0.340
2-Fluorobiphenyl				
Polychlorinated Biphenyls (PCBs) [Method SW846 8082]	mg/Kg		ug/L	
PCB-1016	0.0167	0.00326	0.500	0.176
PCB-1221	0.0167	0.00326	0.500	0.176
PCB-1232	0.0167	0.00326	0.500	0.176
PCB-1242	0.0167	0.00326	0.500	0.176
PCB-1248	0.0167	0.00326	0.500	0.176
PCB-1254	0.0167	0.00782	0.500	0.250
PCB-1260	0.0167	0.00782	0.500	0.250
PCB-1262	0.0167	0.00782	0.500	0.250
PCB-1268	0.0167	0.00782	0.500	0.250
Volatile Organic Compounds (VOCs) [Method SW846 8260]	ug/Kg		ug/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-Trichloroethane	5.00	0.650	1.00	0.230
1,1,2-Trichloro-1,2,2-trifluoroethane	5.00	1.14	1.00	0.310
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-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
Bromoform	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
Dibromochloromethane	5.00	0.640	1.00	0.320
Chloroethane	5.00	1.13	1.00	0.320
Chloroform	5.00	0.309	1.00	0.340

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

	Soil		Groundwater	
	RL	**MDL**	**RL**	**MDL**
Volatile Organic Compounds (VOCs) [Method SW846 8260] (continued)	**ug/Kg**		**ug/L**	
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
Dichlorodifluoromethane	5.00	0.413	1.00	0.680
Ethylbenzene	5.00	0.345	1.00	0.740
1,2-Dibromoethane	5.00	0.642	1.00	0.730
Isopropylbenzene	5.00	0.754	1.00	0.790
Methyl acetate	5.00	0.930	2.50	0.500
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
TCLP Metals [Method SW846 6010]	**mg/L**		**--**	
Aluminum	0.200	0.0600	--	--
Antimony	0.0200	0.00679	--	--
Arsenic	0.0150	0.00555	--	--
Barium	0.00200	0.000700	--	--
Beryllium	0.00200	0.000300	--	--
Boron	0.0200	0.00400	--	--
Cadmium	0.00200	0.000500	--	--
Calcium	0.500	0.100	--	--
Chromium	0.00400	0.00100	--	--
Cobalt	0.00400	0.000630	--	--
Copper	0.0100	0.00160	--	--
Iron	0.0500	0.0193	--	--
Lead	0.0100	0.00300	--	--
Magnesium	0.200	0.0434	--	--
Manganese	0.00300	0.000400	--	--
Nickel	0.0100	0.00126	--	--
Potassium	0.500	0.100	--	--
Selenium	0.0250	0.00870	--	--
Silver	0.00600	0.00170	--	--
Sodium	1.00	0.324	--	--
Thallium	0.0200	0.0102	--	--
Vanadium	0.00500	0.00150	--	--
Zinc	0.0100	0.00150	--	--

Notes:
mg/Kg - milligram per kilogram
ug/Kg - microgram per kilogram
mg/L- milligram per liter
ug/L - microgram per liter

6. REFERENCES

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

EPA Guidance for the Data Quality Objectives Process (EPA-QA/G-4), August 2000.

EPA Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846. Third Edition, November 1986; Revision 1, July 1992; Revision 2, November 1992; Update II, September 1994; Update III, December 1997; and Update IIIA, March 1999.

ATTACHMENT A

TESTAMERICA'S QUALITY ASSURANCE MANUAL AND STANDARD OPERATING PROCEDURES

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Buffalo Quality Memorandum

Date: November 12, 2013

From: Brad Prinzi, Quality Assurance Manager

To: TestAmerica Buffalo

Subject: Lab Quality Manual - Sample Acceptance Policy

The purpose of this memorandum is to update the Sample Acceptance Policy for TestAmerica Buffalo to include our policy for Radiation Screening as outlined in BF-SR-002. Section 23.3 Sample Acceptance Policy will add the following bullet to the acceptance criteria:

- 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 Director, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.

Figure 23-2, as noted below, replaces Figure 23-2 in the Quality Manual.

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 Director, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.

3) 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**

4) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

5) 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).

6) 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.

7) 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.

8) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

9) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.

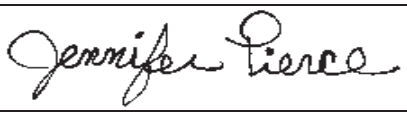
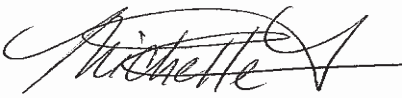
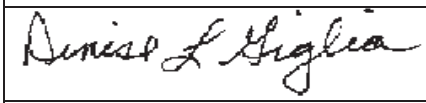
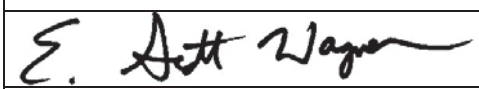
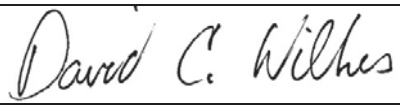
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

   - 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.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Approvals

	11/12/2013
Laboratory Director – Chris Spencer	Date
	11/12/2013
Quality Assurance Manager - Brad Prinzi	Date
	11/12/2013
Operations Manager – Jennifer Pierce	Date
	11/12/2013
Organic Preparation Manager – Michelle Freeman	Date
	11/12/2013
GC/MS Volatiles Manager – Denise Giglia	Date
	11/12/2013
Wet Chemistry Manager – James Rojecki	Date
	11/12/2013
GC Semivolatiles / Volatiles Manager – Gary Rudz	Date
	11/12/2013
Metals Manager – Scott Wagner	Date
	11/12/2013
GC/MS Semivolatiles / IC Manager – David Wilkes	Date

Cover Page:

Quality Assurance Manual

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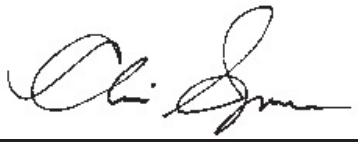
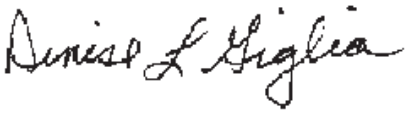


Laboratory Director – Chris Spencer — 02/13/2013 Date

Quality Assurance Manager - Brad Prinzi — 02/13/2013 Date

Operations Manager – Jennifer Pierce — 02/13/2013 Date

Organic Preparation Manager – Michelle Freeman — 02/13/2013 Date



GC/MS Volatiles Manager – Denise Giglia — 02/13/2013 Date

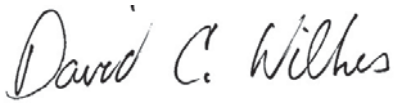
Wet Chemistry Manager – James Rojecki — 02/13/2013 Date

02/13/2013

GC Semivolatiles / Volatiles Manager – Gary Rudz — Date

02/13/2013

Metals Manager – Scott Wagner — Date



02/13/2013

GC/MS Semivolatiles / IC Manager – David Wilkes — Date

SECTION 2

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REFERENCED CORPORATE SOPs AND POLICIES

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-004	Ethics Policy

CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

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 or 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-QA-001	Determination of Method Detection Limits
BF-QA-002	Quality Control Limits
BF-QA-003	Procedure for Writing, Reviewing and Revising Controlled Documents

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., President, Chief Executive Officer, 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 Director, Customer Service 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 based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. 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 Director or Designee

The Technical Director reports directly to the Laboratory Director and is responsible for assessing the construction and management of the facility design, maintaining environmental conditions, technical and financial evaluation of capital equipment and capital budgeting and asset valuation.

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods. Specific responsibilities include but are not limited to:

- 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.
- 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.
- Compliance with ISO 17025 Standard.

4.2.4 Operations Manager

The Operations Manager reports to the Laboratory Director and oversees the daily operations of the analytical laboratory, maintaining a working environment that encourages open, constructive problem solving and continuous improvement.

The Operations Manager is responsible for supervision of laboratory staff, setting goals and objectives for the laboratory, ensuring compliance with project/client requirements and ensuring on-time performance, supervises maintenance of equipment and scheduling of repairs. Responsibilities also include implementation of the quality system in the laboratory and ensuring timely compliance with audit and QA corrective actions.

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment, assessing capital budget needs and determining the most efficient instrument utilization. More specifically he:

- 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 Director and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- 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 Director, 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 Director, 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 Environmental Health & Safety / Hazardous Waste Coordinator

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste and preparation of Safety related SOPs. The EHSC maintains overall EH&S program oversight, but may delegate specific day-to-day activities as necessary.

- 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.
- 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 Material Safety Data Sheet (MSDS) 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 Test America’s medical consultants.

4.2.7 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.

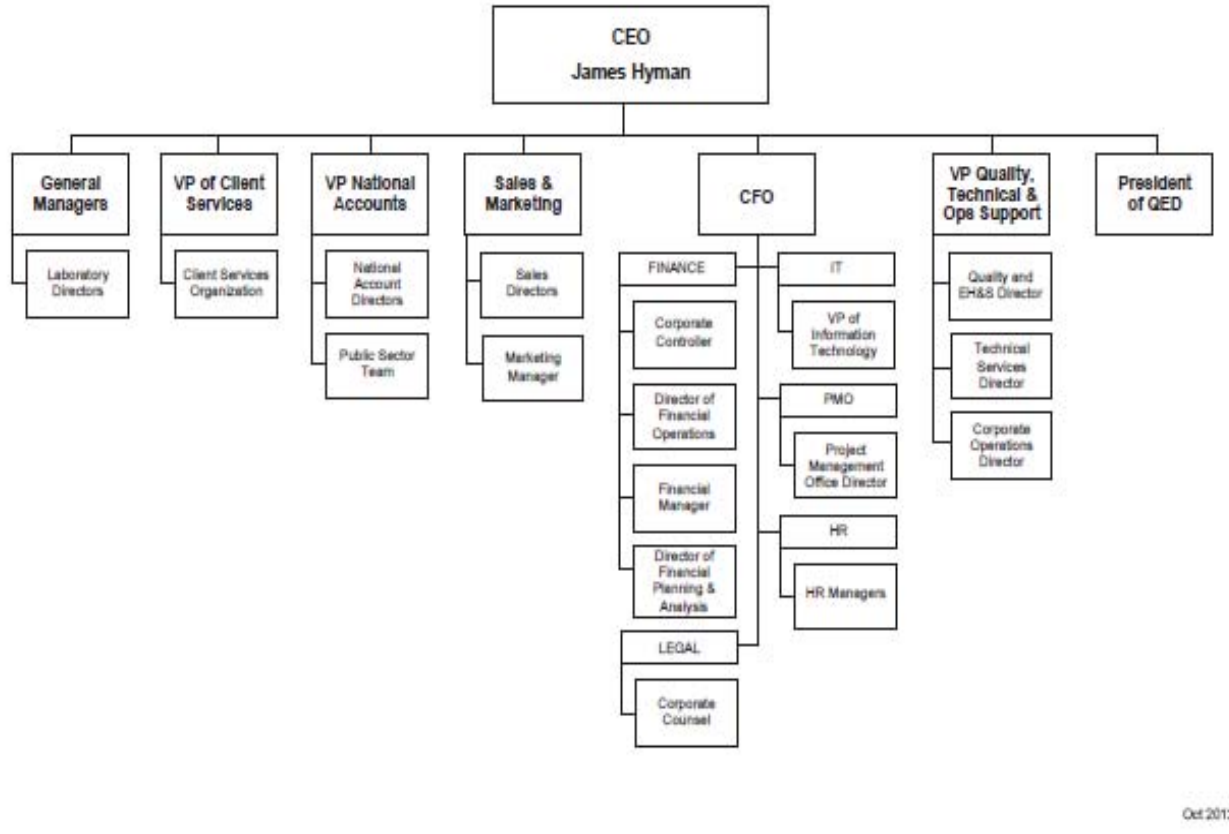
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, 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:

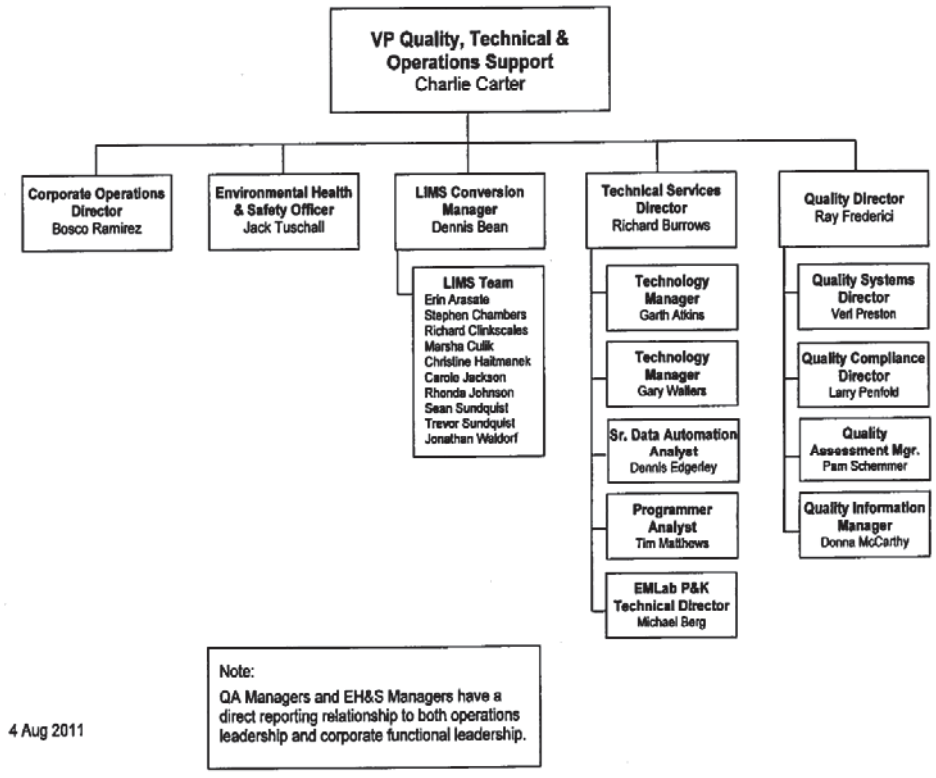
Key Personnel	Deputy	Comment
Laboratory Director	Operations Manager (1) Technical Director (2)	
QA Manager	QA Specialist (1) Operations Manager (2)	
Technical Director	Laboratory Director (1) Operations Manager (2)	
Operations Manager	Department Manager (1) Department Manager (2)	Selected based on availability
Customer Service Manager	Project Mng't Manager (1) Laboratory Director (2)	
Project Management Manager	Customer Srv. Manager (1) Project Manager (2)	(2) Selected based on availability
Project Manager	Project Manager (1) Project Management Asst. (2)	(1) 2° 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	Safety Officer (1) Sample Mng't 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



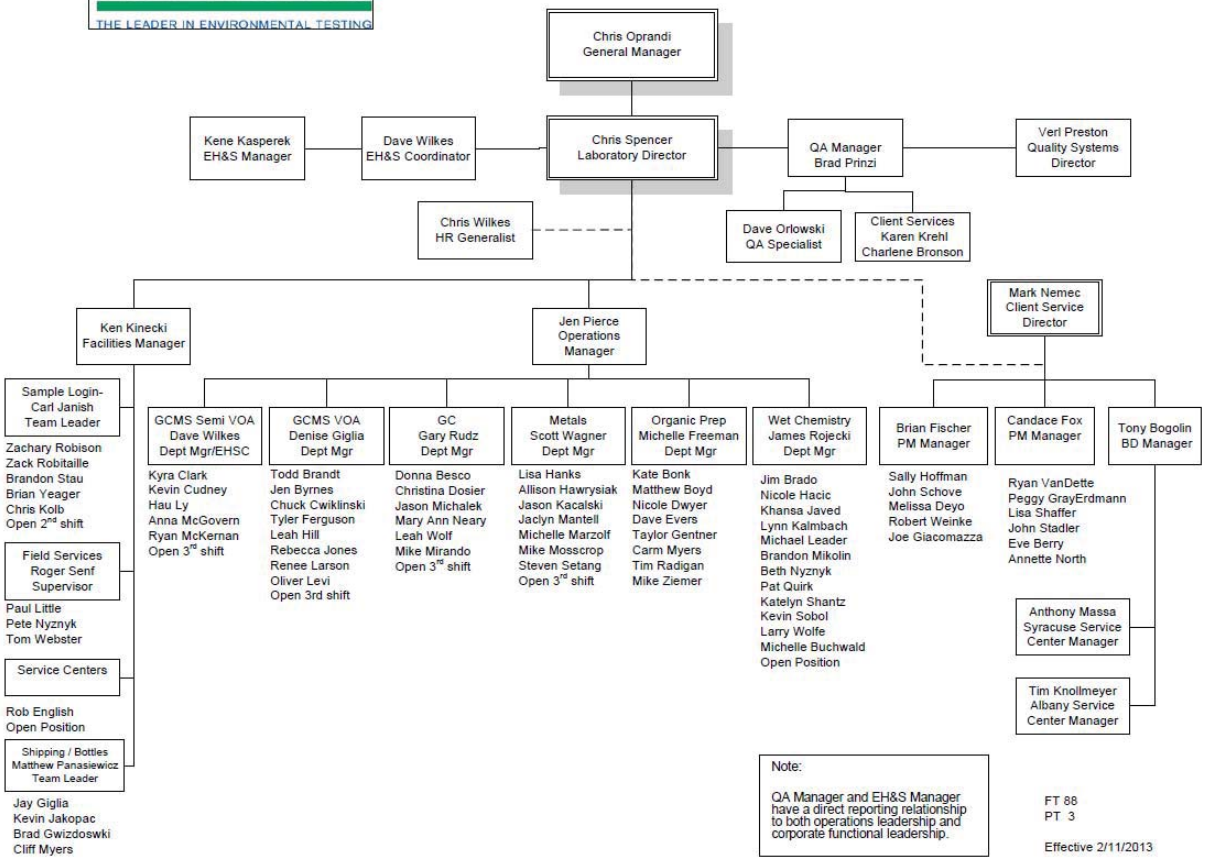
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Quality, Technical & Operations Support



4 Aug 2011

Buffalo Laboratory Organization



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
-

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 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 and other management. 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 and every 1 year for Drinking Water programs. 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 in the QA office. Electronic versions are kept in a controlled access electronic folder in the QA department. As revisions are required, a new version number and revision date is assigned and the document placed on the laboratory Intranet (BufNet) for use.

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 National Account Director, 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):

- Legal & Contracts Director
- General Manager
- Customer Service Manager
- Operations Manager
- Laboratory and/or Corporate Technical Directors
- 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 National Account Director, Legal Contracts Director, or local account representative 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 Legal & Contracts Director maintains copies of all signed contracts. The Customer Service Manager 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 Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Customer Service Manager.

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, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively 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).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representative's cooperation to clarify the client's request". This topic is discussed in Section 7.

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 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) and the Work Sharing Process SOP (CA-C-S-001).

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), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing 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.

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, Regional Account Executive (RAE) or Customer 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. (Corporate SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, then to begin the process, 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 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 Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group 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 Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE 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, facilities 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 Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007

Contracts will be signed in accordance with TestAmerica's Corporate 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 coordinator 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. Material Safety Data Sheets (MSDSs) 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^{o}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 Director 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 Director 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 Director.

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 Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and 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 Director 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 following the laboratory SOPs related to Data Quality Review (BF-QA-006) and 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 SOPs BF-QA-006 and 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 QA 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 Director, 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 job exception 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 Director, 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 Director, the Operations Manager or the QA Manager 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 job exception 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 Director, Operations Manager, QA Manager, Customer Service Manager, Human Resources Manager and Business Development 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 & Client Advocacy 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, General Managers 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.

On a monthly basis, 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 General Manager 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 Director, 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 Customer Service Manager 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 Report (NCR) also know as Job Exception Reports (JER) 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 Report (NCR) - (previously known as Job Exception Report and Data Quality Review (DQR) - 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 JERs.
- Issues found while reviewing JERs that warrant further investigation.
- Questionable trends that are found in the monthly review of DQRs or client complaints

- 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 NCR 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 Director, 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 NCR 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 NCR and DQR are entered into a database and each CAR is entered into a spreadsheet for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCR 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 NCR 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 NCR 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

TA Corrective Action Summary

Corrective Action Summary

Rev. 0

#	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	28-Jan-13	Follow-up Closed By
1																			
2																			
3																			
4																			
5																			
6																			
7																			
8																			
9																			
10																			
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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 [1]	- 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 Director, 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.

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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, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluations of proficiency testing (PT) performance, 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. 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.

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 provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.

- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review

13.1.2 Any Preventive Actions 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 past 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 Data Deliverables Manager while electronic technical records are maintained by the IT Administrator.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - 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 Types [1]:	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 in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Retention of records is maintained on-site at the laboratory for at least 3 months after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless other wise 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.

Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

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

[1]Note: 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 All records are held secure and in confidence. Records maintained at the laboratory are located in the locked on-site storage room. Records archived off-site are stored in a secure location. Access to the off-site storage facility is controlled and logs are maintained for the documented removal/return of records

14.1.4 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.

14.1.5 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 (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.

14.5.7.3 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-004)	Methods Audits Frequency: 50% of methods annually
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

* = all methods receive a QA Technical Audit or an SOP Method Compliance Audit annually.

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, NELAC 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 Director 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, Nonpotable 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 for review and comments. The final report shall be submitted to the Operation Manager as well as the appropriate Quality Director and General Manager. 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 General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Director, Operations Manager, Customer Service 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-008 & Work Instruction No. CA-Q-WI-020) 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 General Manager 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, General Managers and Quality Directors receive a monthly report from the Corporate Quality Director summarizing any current data integrity or data recall investigations. The General Manager's 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 Buffalo Human Resource office (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 Directors/Department Managers – **General**	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 ft^2 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, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *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 / 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*

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.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager 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 repreparation (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 $\pm$ 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 (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/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"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/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 out lined 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 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Project Managers perform review of the chain-of-custody forms and inputted information and approve the input in LIMs to make the samples available to the laboratory departments for batching and processing.

19.14.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add any manual data qualifiers or dilution codes if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 10% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. 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

- Results deviate from historical trends (if history available)

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any unusual or uncharacteristic circumstances are brought to the attention of the Department Manager. The Department Manager may involve the Project Manager, the Technical Director and/or the QA Manager for further investigation depending on the issue. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.5 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.

19.14.4.6 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 principals 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

DOC Cert. Statement
Revision 11
January 23, 2013

TESTAMERICA LABORATORIES, INC.

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

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.

Jennifer Pierce _____ _____
Operations Manager Signature Date

_____ _____ _____
Quality Assurance Officer 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.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) 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 Repipetters" and BF-GP-002, "Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devises 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 $\pm$ 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 annually. 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 > 0ºC and $\leq$ 6 ºC.

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.

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 ever 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

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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	Clarus 608 dual uECD	680S10042901	2012	good
GC Instrumentation	Perkin Elmer	Clarus 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 Hall/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	Konelab	Aqua20	SEA032	2009	good
Water Quality Instrumentation	Flash Point Analyzer		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 Magic - Autoanalyzer	270H3XB531	2004	good
Water Quality Instrumentation	Labtronics	BOD Magic - Autoanalyzer	270J2XB669	2003	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	OI	Carbon Analyzer Model 1010 #1	H92170411	1999	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	Orion	Ion Meter #230A	2229	1999	good
Water Quality Instrumentation	VWR	Ion Meter #2100	1063	1997	good
Water Quality Instrumentation	YSI	Oxygen Meter #57	93J09826	1995	good
Water Quality Instrumentation	BOD chamber		Revco	1994	good
Sample Preparation Equipment	TurboVap	II	TV0529N12427	2006	good
Sample Preparation Equipment	TurboVap	II	TV0529N12428	2006	good
Sample Preparation Equipment	J2	ACCUPREP GPC	03F-10723	2003	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
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

Instrument	Procedure	Frequency
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Vacuum Pumps/ Air Compressor	Drained	Weekly
	Belts checked	Monthly
	Lubricated	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	± 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	± 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	± 1.2°C	Replace
Minimum-Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 1.5°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	± 1.5°C	Repair/replace
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	0-6°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)°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 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°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	± 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.	± 1%	Not applicable.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Daily	<1.0 µmho at 25°C	Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Director.

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. 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 A2LA or NVLAP with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The 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.

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 raw data.

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-002	Sample Packaging and Shipment Off-Site
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. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

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. The

first day of holding time for time critical parameters ends twenty-four hours after sampling. 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 following tables 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.

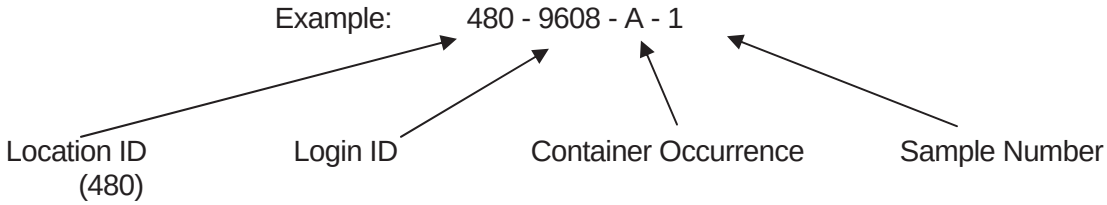
23.2.1 Laboratory Receipt

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);
- 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)

[redacted]

Chain of Custody Record

Temperature on Receipt _____

Drinking Water? Yes☐ No☐

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TAL-4124 (1007)

Client	Project Manager	Date	Chain of Custody Number 193232
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact / Lab Contact	Analysis (Attach list if more space is needed)	Special Instructions/ Conditions of Receipt
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	Air	Aqueous	Sed.	Soil	Unpres.	H2SO4	HNO3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
(A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

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) 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**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) 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).

5) 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.

6) 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.

7) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

8) 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

Doc. Login Front

TestAmerica Buffalo

Doc. LoginFront
Rev 13
09/21/2012

LOGIN **Copy from**

Company_____ Project #_____

Event _____ Analysis_____

TAT_____BD/_____CD # OF SAMPLES _____ TRIP BLANK Y/N #_____

SHIPPED BY	ATTACH SHIPPING TAGS(back)
RECEIVED DATE / TIME:	_____/_____/_____ _____: _____

COOLER(s)_____ Temps_____ °C (<6 °C)

IR GUN 1 2 3

CUSTODY SEAL INTACT? YES/NO NONE RAD CHECK <0.02mR/hr: Y/N

RESIDUAL CHLORINE CHECK

☐ YES, OK ☐ YES, Qualified ☐ YES, Preserved ☐ NO, lab to check ☐ N/A

WORKSHARE/SUB Y/N LAB_____ ICOC #_____

RECEIVED OUTSIDE HOLD TIME Y/N_____

CHECKLIST ISSUES Y/N_____

PRESERVATION CHECKED YES___ NO____ NA____ Initials_____

ARE SAMPLE DATES AND TIMES CORRECT? Initials_____

WERE ALL THE APPROPRIATE TESTS ASSIGNED? Initials_____

NCM_____ COC SCANNED Y/N

ANALYSIS GROUP ISSUES_____

Temp Cert Loss:

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 qualify 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 affects 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). 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 $\pm$ 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 $\leq$ 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.

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.

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.
- In most cases, the applicable COC is paginated and is an integral part of the report.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).

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 to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.19 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.

25.2.20 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.

25.2.21 The laboratory includes a cover letter.

25.2.22 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.

25.2.23 When Soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.24 Appropriate laboratory certification number for the state of origin of the sample if applicable.

25.2.25 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.

25.2.26 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.

25.3 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. ***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.

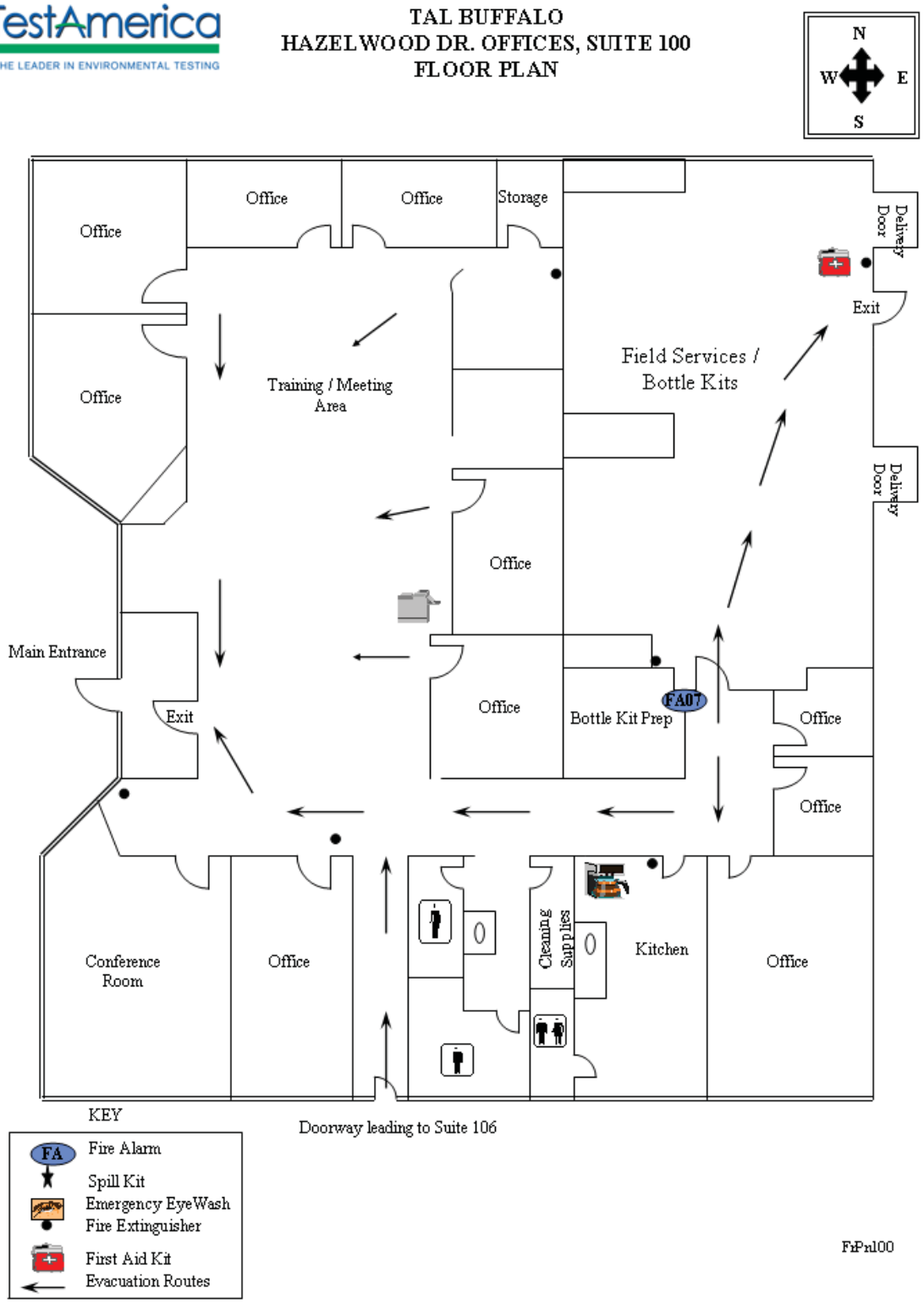
25.9.2 Multiple Reports

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.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 100
FLOOR PLAN



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 106
CLIENT SERVICES/REPORT PREP
FLOOR PLAN

FrPl106L
3/2005

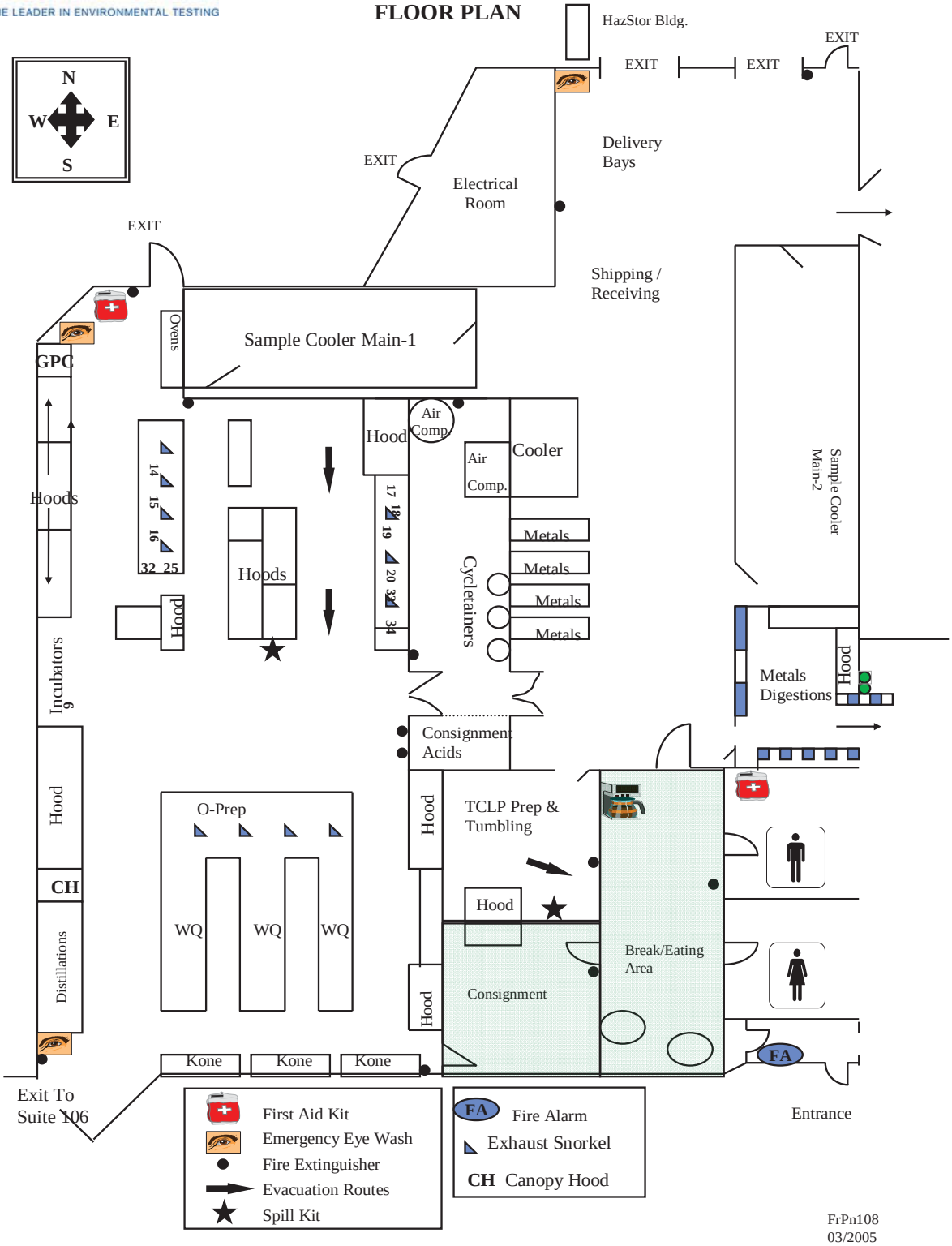
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. NY OFFICES, SUITE 106
LABORATORY AREA
FLOOR PLAN

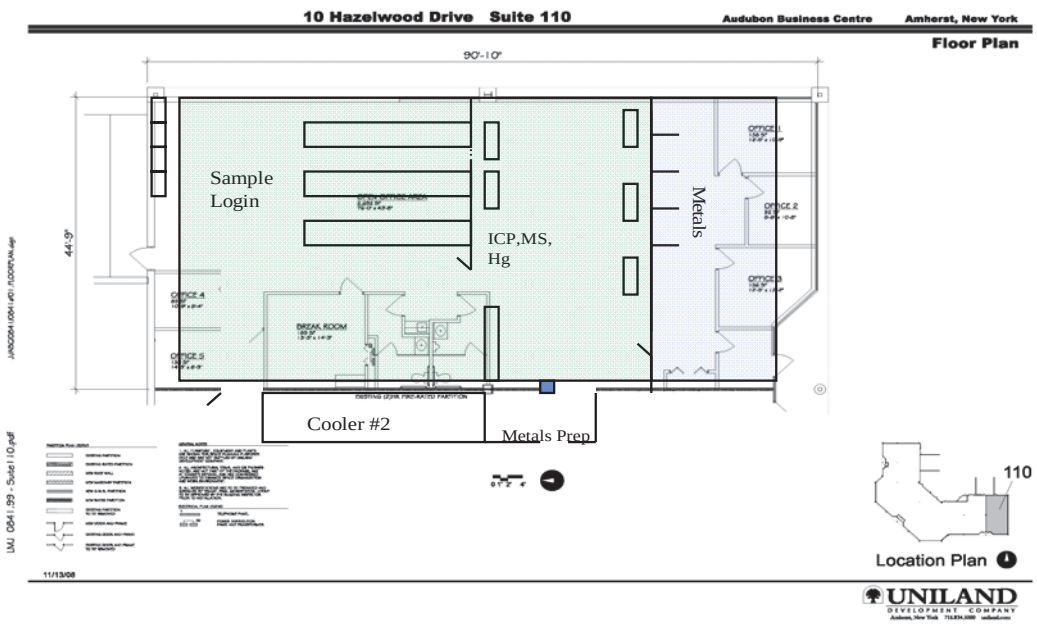
FrPn106r
03/2005

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 108
FLOOR PLAN



FrPn108
03/2005


10 Hazelwood Drive Suite 110
Audubon Business Centre
Amherst, New York
Floor Plan
90'-10"
44'-9"
Sample Login
ICP,MS, Hg
Metals
OFFICE 1
OFFICE 2
OFFICE 3
OFFICE 4
OFFICE 5
BREAK ROOM
Cooler #2
Metals Prep
110
Location Plan
11/13/08
UNILAND
DEVELOPMENT COMPANY

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)

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

Derivitization
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 re 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)

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 if 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 filing 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 ± 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 sorbant 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.

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 2^{nd} 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 2^{nd} 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
MSDS - Material Safety Data Sheet
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
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*	*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 parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, and in the QA Department.

Title: n-Hexane Extractable Material (HEM) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM) by Solids Phase Extraction Method No. 1664

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

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Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method is taken from EPA method 1664 revision A, which is applicable to waters and soils. This method is applicable to surface and saline waters as well as industrial and domestic aqueous wastes. This method covers a range from 5 to 1000 mg/L of extractable material.

2.0 Summary of Method

2.1 One liter of sample is acidified to a low pH (below 2.0), then filtered through a disk, which has been conditioned with n-Hexane and Methanol. The Oil and Grease is then extracted from this disk using Hexane. This solvent is evaporated using a Speed Vap and the remaining residue is weighed.

2.2 If the HEM is to be used for determination of SGT-HEM, the HEM is redissolved in n-hexane.

2.3 For SGT-HEM determination, an amount of silica gel proportionate to the amount of HEM is added to remove polar materials. The solution is filtered to remove the silica gel, the solvent is evaporated, and the SGT-HEM residue is weighed.

3.0 Definitions

3.1 The definition of oil and grease is based on the procedure used, the nature of the oil/grease and the presence of extractable non-oily matter that will influence the material measured and interpretation of the results.

3.2 PAR Standard: Precision and Accuracy solution

3.3 HEM: Hexane Extractable Material

3.4 SGT-HEM: Silica Gel Treated Hexane Extractable Material

3.5 Standard definitions are in section 3.0 of the Laboratory Quality Manual.

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts that can affect results. Specific selection of reagents and purification of solvents may be required.

4.2 All material used in the analyses shall be demonstrated to be free from interference by running laboratory blanks.

4.3 Glassware is cleaned by rinsing with solvents mentioned in section 7.0 of this SOP, by washing in hot water containing detergent, and finally rinsing with distilled water.

4.4 Interference extracted from samples will vary considerably from source to source, depending on the diversity of the waste being analyzed.

4.5 Water must be completely removed from the extract prior to evaporation. On humid days, water may condense in the extract during evaporation, in which case further evaporation or low temperature heating may be required.

4.6 Sodium sulfate and silica gel may on occasion come through the final filter. A smaller pore size filter must then be used.

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 n-Hexane has been shown to increase neurotoxic effects over other hexanes and some other solvents. Inhalation of n-hexane should be minimized by performing all operations with n-hexane in an explosion proof hood or well-ventilated area.

5.1.2 n-Hexane has a flash point of -23 C (-9.4 F), has explosive limits in air in the range of 1-7 percent, and pose a serious fire risk when heated or exposed to flame. n-Hexane can react vigorously with oxidizing materials.

5.1.3 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

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
Hexane	Flammable Irritant	500 PPM-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 PPM-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
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 Equipment

- Solids phase extraction manifold with pump and trap.
- Analytical balance
- Speed Vap II solvent evaporation system, manufactured by Horizon

6.2 Supplies

- Dessicator
- Solid phase extraction disks (manufactured by CPI)
- Oil/Grease weigh tins, purchased from Horizon
- pH paper
- 10 ml volumetric pipette
- 1000ml and 2000ml graduated cylinder
- 1 PS phase separator paper
- Vials, with PTFE lined screw caps (40 ml)
- Magnetic stir plate
- PTFE coated magnetic stir bars

- Fastflo prefilters, optional (used for samples high in suspended solids)

7.0 Reagents and Standards

7.1 n-Hexane- 85% minimum purity, 99% minimum saturated C6 isomers, residue <1 mg/L.

7.2 Methanol ACS grade

7.3 Sodium Sulfate Anhydrous (granular only)

7.4 Two sources of Oil & Grease Standard (2mg n-hexadecane, 2mg stearic acid); pre-purchased from CPI and Environmental Express. The solution should be checked frequently for signs of degradation and evaporation. Keep tightly capped and store in the reagent refrigerator. Bring solution up to room temperature for 1 hour to ensure that stearic acid is in solution.

7.5 1+1 Hydrochloric Acid

7.6 Silica gel - anhydrous, 75-150 micrometers, dried at 200-250 ºC for 24 hour minimum and stored in a dessicator or tightly sealed container. Determine the n-hexane soluble material content of the silica gel by extracting 30 g of silica gel with n-hexane and evaporating to dryness. The silica gel must contain less than 5 mg of n-hexane soluble material per 30 g.

7.7 Laboratory reagent water

7.8 Nitrogen source

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Glass	1000-mL	HCl, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soils	Glass	5 grams	Cool 4 ± 2°C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Laboratory Control Sample (LCS): (HEM =40.0mg/L, SGT-HEM =HEM recovery/2). Prepare a LCS standard by filling a 1000ml-graduated cylinder with DI water and pipette 2.0ml of 1+1 Hydrochloric acid. Pour this into the 1000ml bell

and add the entire contents of a 5mL Restek Oil and Grease standard (Restek catalog # 31457). The true value is 40.0mg/Lfor HEM analysis; the true value for SGT-HEM analysis is obtained by dividing the recovery for HEM analysis by 2 to reflect the concentration of hexadecane that remains after removal of stearic acid. Analyze one LCS every 20 samples. If an acceptance criteria is exceeded all samples bracketed must be repeated.

9.1.2 Method Blank (MB): Freedom from contamination is determined by analyzing a method blank every 20 samples. Extract and concentrate one liter of laboratory reagent water carried through the entire analytical procedure. The method blank must exhibit values less than the reporting limit (5.0 mg/L).

9.1.3 Matrix Spike (MS) / Matrix Spike Blank (MSB): (HEM=20.0mg/L, SGT-HEM =HEM recovery/2). Prepare MS/MSB by pipetting 5.0ml of the Stock PAR standard into a laboratory sample to create the MS, or into 1 liter reagent grade water to create an MSB. The true value is 20.0mg/L for HEM analysis; the true value for SGT-HEM analysis is obtained by dividing the recovery for HEM analysis by 2 to reflect the concentration of hexadecane that remains after removal of stearic acid. Analyze one MSB per 20 samples. If an acceptance criteria is exceeded all samples bracketed must be repeated. A Matrix Spike Blank is only required if an extra volume is not present for a Matrix Spike.

9.1.5 LCS, MS and MSB acceptance criteria for HEM are 78-114 % and for SGT-HEM the acceptance criteria are 64-132%.

9.1.6 Soil Laboratory Control Sample (LCS): LCS is purchased from Environmental Resource Associates (ERA). A soil LCS is run every 20 or fewer samples. The "True Value" for standards purchased from ERA varies depending on the Lot number. All ERA Certificate of Analysis are kept on file for easy reference.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Waters	1000-mL of sample or dilution
Soils/Wastes	5 grams

10.2 Calibration

10.2.1 Analytical balances are calibrated every 6 months and checked daily to ensure calibration is maintained.

10.2.1.2 Prior to analysis the calibration of the balance needs to be verified at 2mg and 1000mg. The values need to be ±10% at 2mg and ±0.5% at 1000mg. If the values are not within these limits, recalibrate the balance before proceeding with analysis. Upon completion of analysis this same procedure needs to be followed to re-verify the calibration. All weights will be recorded directly into the LIMS system.

10.2.2 If there is doubt of the concentration of the PAR solution, remove 10.0 ± 0.1 ml with a volumetric pipette, place in a tarred weighing pan, and evaporate to dryness in a fume hood. The weight must be 40 ± 1 mg. If not does not meet this criteria discard standard and open a new bottle.

10.3 Aqueous Sample Analysis

10.3.1 Bring the samples to room temperature.

10.3.2 Verify the pH of the sample is less than 2. Dip a glass-stirring rod or the bulb of a disposable pipette into the sample, withdraw the stirring rod/pipette and allow a drop of sample to fall on a touch pH paper. Adjust accordingly if needed.

10.3.3 Mark the sample bottle at the water meniscus for later determination of the sample volume.

10.3.4 If the sample is high in suspended solids, allow solids to settle or use a fastflo prefilter. When adding the sample, tilt the sample container to allow particulates to settle on one side. This is suggested to aid in decanting of the liquid portion of the sample. If sample is extremely high in settable solids split the sample into two bells and combine hexane extracts into one weigh tin.

10.3.5 Extraction Disk Conditioning:

NOTE: Proper disk conditioning is critical for a successful extraction. Failure to condition the disk properly may result in erratic and/or low recoveries.

10.3.5.1 Place the disk on the manifold, ripple side up. If using fastflo prefilters, place in the bottom of the reservoir, 1 mm recessed from the bottom edge of the reservoir. Fill in spaces with glass wool if necessary.

10.3.5.2 Wash the disk with 10 ml (25 ml if using fastflo prefilters) of n-hexane. NOTE: Always run solvents down the sides of the glassware when washing and eluting. Allow the disk to soak for 2 minutes.

10.3.5.3 Apply vacuum to pull remaining solvent through the disk. Allow the disk to dry.

10.3.5.4 Repeat steps 10.3.5.2 and 10.3.5.3.

10.3.5.5 Add 10 ml (25ml if using fastflo prefilters) of methanol to the reservoir. Apply a light vacuum and pull approximately 1 ml through the disk. Allow the disk to soak for 1 minute. Add an additional 10mls (25mls if using fastflo prefilters) of methanol to the reservoir. Apply a light vacuum and pull approximately 1ml of solvent through the disc. Allow the disc to soak for 1 minute. **It is critical that the disc is not allowed to go dry during this stage of conditioning.**

10.3.5.6 Add 10 ml of reagent grade water to the reservoir. Apply a light vacuum and pull the reagent grade water through the disk until the surface is covered with a 1-2 mm of water. Allow the disk to soak for 2 minutes.

****NOTE**: The extraction disk should be brought to near dryness during this step, but it is important that the disk is not allowed to dry before introducing the sample. The sample must not come into contact with residual methanol. Drying of the disk could lead to decreased yields**

10.3.6 Sample Extraction:

10.3.6.1 Verify that no residual methanol is remaining on the filter disk, then pour or decant the sample into reservoir and apply vacuum. Decant and extract as much liquid as possible before adding sediment to the reservoir. Do not let the disk go dry before adding sediment. After sample has been poured off into the sample reservoir (bell) for analysis, add 30mL of hexane to the sample container, cap, shake to rinse all the surfaces inside the sample container. Collect the rinsate in the same reservoir being used for the sample analysis.

10.3.6.2 After sample extraction is complete, remove as much residual water as possible from the disk by applying vacuum to dry the disk for 10 minutes.

10.3.7 Sample Elution:

NOTE: Using an extraction manifold outfitted with 40-mL VOA vials**

10.3.7.1 Weigh out weigh dishes and record in the designated Excel spreadsheet.

10.3.7.2 Elute with 10-mL of Hexane into vial. Smaller volumes of solvent may be used once elution techniques have been perfected. When adding elution solvent, rinse down the sidewalls of the reservoir and the sample bottle. Allow the n-hexane to soak the disk for 2 minutes before applying vacuum.

10.3.7.3 Repeat the process in steps 10.3.8.2 with a second aliquot of eluting solvent.

10.3.7.4 Transfer eluted solvent to weighing tin on Speed Vap II. Rinsing the vial used in elution process with n-hexane and adding the rinsate to weighing tin ensures that extracted material is not lost in the transfer process.

10.3.7.5 To determine the original sample volume in liters fill the sample bottle to the mark with water and measure the volume of the water in a 2 liter graduated cylinder and record the volume.

10.3.7.6 Evaporate the n-hexane in a Speed Vap II system. The temperature of the Speed Vap II can be adjusted to decrease evaporation time, but should not exceed 47 degrees Celsius. If a significant amount of water is present after elution, a filtration using a Whatman 1PS Phase separating silicone treated filter paper can be used to remove the water.

NOTE: Pre-rinse the filter with solvent to remove trace amounts of silicone.

10.3.7.7 Purge the weighing tin with a gentle stream of nitrogen to ensure that all solvent has been evaporated off, and to remove any water that may have condensed

during the evaporation process. Place weighing tins in the desiccator until stable temperature is obtained.

10.3.7.8 Weigh the dishes until a constant weight is obtained. To ensure that a constant weight has been reached, the differences between the readings should not vary by more than 0.0005 grams.

10.3.8 PROCEDURE FOR SGT-HEM:

10.3.8.1 Add 3.0 ± 0.3 g of anhydrous silica gel to a beaker for every 100 mg of HEM, or fraction thereof, to a maximum of 30 g of silica gel. Re-elute HEM with n-hexane, using enough solvent to ensure that all HEM is re-dissolved. Combine HEM solution with silica gel. Stir the solution on a magnetic stirrer for a minimum of 5 minutes.

10.3.8.2 Filter the solution through n-hexane moistened filter paper into a pre-weighed weigh tin. Rinse the silica gel and filter paper with several small amounts of n-hexane to complete the transfer.

10.3.8.3 Evaporate the n-hexane in a Speed Vap II system. The temperature of the Speed Vap II can be adjusted to decrease evaporation time, but should not exceed 47 degrees Celsius. If a significant amount of water is present after elution, a filtration using a Whatman 1PS Phase separating silicone treated filter paper can be used to remove the water.

NOTE: Pre-rinse the filter with solvent to remove trace amounts of silicone.

10.3.8.4 Purge the weighing tin with a gentle stream of nitrogen to ensure that all solvent has been evaporated off, and to remove any water that may have condensed during the evaporation process. Place weighing tins in the desiccator until stable temperature is obtained.

10.3.8.5 Weigh the dishes until a constant weight is obtained. To ensure that a constant weight has been reached, the differences between the readings should not vary by more than 0.0005 grams.

10.4 PROCEDURE FOR EXTRACTION OF SOLID/SLUDGE SAMPLES

10.4.1 Carefully homogenize sample, thoroughly mixing any water that may have settled on the top of the sample.

10.4.2 Weigh 5 grams of homogenized sample into a clean 40-mL VOA vial. Record exact weight of sample used on spreadsheet.

10.4.3 Add approximately 5 grams of granular, purified sodium sulfate to vials. Mix sample and sodium sulfate well. If SGT only is requested add 3 grams of silica gel.

****NOTE:** To prepare the Method Blank (MB) add approximately 5 grams of granular, purified sodium sulfate to a clean 40mL VOA vial and record the exact weight as the sample weight.

To prepare the LCS for HEM add approximately 5 grams of ERA soil standard #1 to a clear 40 mL VOA vial and add approximately 5 grams of granular, purified sodium sulfate to the vial. Mix sample and sodium sulfate well. Record exact weight of sample used on spreadsheet. Repeat these steps to prepare a LCS for SGT – HEM but replace ERA standard #1 with ERA standard #2.

10.4.4 Add 15-mL of n-hexane to vial and immediately cap. The solvent should completely cover the solids. Gently invert vial to ensure that any lumps that may have formed are broken up.

10.4.5 Place vials in the sonicator ensuring that the water level of the sonicator is well above the solids and hexane. Sonicate vials for 3 minutes.

10.4.6 Collect the extract in another clean 40-mL VOA vial by filtering extract through a 1 PS phase separator paper pre-moistened with n-hexane.

10.4.7 Repeat steps 10.4.4 - 10.4.6 two more times.

10.4.8 Evaporate the n-hexane in a Speed Vap II system. The temperature of the Speed Vap II can be adjusted to decrease evaporation time, but should not exceed 47 degrees Celsius.

10.4.9 Purge the weighing tin with a gentle stream of nitrogen to ensure that all solvent has been evaporated off, and to remove any water that may have condensed during the evaporation process. Place weighing tins in the desiccator until stable temperature is obtained.

10.4.10 Weigh the dishes until a constant weight is obtained. To ensure that a constant weight has been reached, the differences between the readings should not vary by more than 0.0005 grams.

10.4.11 If in addition to HEM, an SGT-HEM analysis is requested for the solid/sludge sample, re-elute the HEM with n-hexane and follow Section 10.3.9, the procedure for SGT-HEM.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\textbf{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration =

$$\text{O\&G mg/L} = \frac{\text{Weight of Tin + Sample (g) - Weight of Tin (g)}}{\text{Sample volume (ml)}} \times 10{,}000$$

$$\text{O\&G mg/kg} = \frac{(\text{ Weight of Tin + Sample (g) – Weight of Tin (G) X 1000 })}{\text{Sample weight (g)}}$$

These calculations are done automatically in the Excel spreadsheet

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. 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 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 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.

12.3 Training Requirements

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.

The following analyst validation information is maintained for this method in the laboratory QA files.

- ✓ The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- ✓ The analyst must read and understand this SOP.
- ✓ The analyst must read and understand the Method used as reference for this SOP.
- ✓ The analyst must complete a DOC or successfully analyze PT samples annually.
- ✓ 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. The following waste streams are produced when this method is carried out.

- Acidic waste generated by the analysis. Acidic waste generated by the analysis is to be disposed of in "A" waste**.**
- Solvent waste. Solvent waste should be disposed of in "C" waste. This includes all rinsing solvents used for cleaning glassware.
- Contaminated disposable glassware and contaminated filter paper generated in the laboratory. Contaminated disposable glassware is to be disposed of in the recycling bin. Contaminated filter paper is to be disposed of in BC waste.

15.0 References / Cross-References

15.1 EPA Office of Water, 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 Gravimeter

15.2 Test Methods for Evaluation Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC; Update III, September 1994.

16.0 Method Modifications:

Item	Method	Modification
10.4.1	*9071A*	*Any water that may have settled is mixed back into sample, rather than decanted, to ensure homogenization of sample*
10.4.5-10.4.8	*9071A*	*Samples are sonicated for 3 minutes and put on a Speed Vap II system rather than extracted in Soxhlet.*

17.0 Attachments

17.1 Analytical Sequence
17.2 Analytical Batch-Aqueous
17.3 Analytical Batch-Solid
17.4 Wet Chemistry Batch Summary

18.0 Revision History

- Revision 4, dated 2 August 2013
    - Updated *Approvals/Signature* section.
    - Updated sections 9.1.1 and 9.1.3 as they were incorrect
    - Updated section 10.4.3 to include more information on how to prepare the standards for soil oil and grease.
    - Updated 10.3.6.7 and 10.3.8.3 in include phase separating filters to remove residual water
    - Updated 10.4.6 to add rinse of filtration paper with solvent is necessary.

- Revision 3, dated 21 December 2012
    - Updated *Approvals/Signature* section.
    - Deleted Section 7.2; Acetone is no longer used for this procedure
    - Update Section 10.3.5.6; Added notation that samples should not come into contact with methanol during the extraction (filtration) step
    - Deleted Section 10.3.7; Sample drying using acetone is no longer used for this procedure.

- Revision 2, dated 15 March 2012
    - Changed Quality Manager, signature added.
    - Added Sect 10.2.1.2 outlining proper verification of balance calibration

- Revision 1, dated 15 April 2009
    - Integration for TestAmerica operations
    - Update Department and QA manager names and signatures
    - Update Attachments 17.2, 17.3, 17.4

Attachment 17.1
Analytical Sequence

LCS
BLANK
MS or MSB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample

Attachment 17.2
Analytical Batch-Aqueous

Laboratory Bench Sheet
Oil Grease
Revision 2 - November 2007

TestAmerica - Buffalo

Analyst:	JFR/JME		**LCS (CHK1) Information:**					**MSB (CHK2) Information:**			**BATCH #:**	**9D11005**
Start Date:	4/10/2009		Lot #		9010905	Filter Lot#	30905	Lot #			9010266	
Start Time:	10:15		Prep Date:					Prep Date:				
End Date:	4/11/2009		Concentration (mg/L)					Concentration (mg/L):				
End Time:	1:10		Expiration Date:					Expiration Date:				
			LCS (CHK1)	True value:		25		MSB(CHK2) Information:		True value	20	
							SOLUTIONS:	Hexane	CHB-46-D			
			RV:	1.0	mg/L			Acetone				
			EQL:	5.0	mg/L			Methanol	CHB-46-B			
Job#	Sample ID	Flask	Spiked (Y/N)	Sample Amount (mL)	Pre-wt. (g)	# 1 Post Wt (g)	# 2 Post Wt (g)	# 3 Post Wt (g)	Calculated DF Factor	Post wt-Pre wt (mg)	Final Conc. (mg/L)	% Rec.
CHK1	LCS	1	Y	1000	6.4111	6.4306	6.4307		1.00	19.6	19.6	78.40%
CHK1	BLANK	2		1000	6.4609	6.4566	6.4568		1.00	-4.1	ND	#VALUE!
CHK2	MSB	3	Y	1000	6.4102	6.4258	6.4259		1.00	15.7	15.7	78.50%
rsd0368	01	4		1000	6.4065	6.4070	6.4066		1.00	0.1	ND	
rsd0357	01	5		970	6.4012	6.3994	6.3998		1.03	-1.4	ND	
rsd0380	01	6		920	6.3867	6.3871	6.3871		1.09	0.4	ND	
rsd0382	01	7		820	6.4393	6.4433	6.4434		1.22	4.1	5.0	
rsd0407	01	8		970	6.3771	6.3753	6.3752		1.03	-1.9	ND	
rsd0408	01	9		960	6.4161	6.4143	6.4140		1.04	-2.1	ND	
	02	10		960	6.4024	6.4020	6.4025		1.04	0.1	ND	
	03	11		990	6.3715	6.3712	6.3710		1.01	-0.5	ND	
	04	12		970	6.4266	6.4265	6.4261		1.03	-0.5	ND	
	05	13		980	6.4315	6.4302	6.4301		1.02	-1.4	ND	
									#DIV/0!	0.0	ND	
CHK1	LCS	33	Y	1000	6.4278	6.4507	6.4507		1.00	22.9	22.9	92%
CHK1	BLANK	34		1000	6.4222	6.4206	6.4211		1.00	-1.1	ND	#VALUE!

Page 1 of 8

Attachment 17.3
Analytical Batch-Solid

Laboratory Bench Sheet
SOIL Oil and Grease
Rev 1 - November 2007

TestAmerica - Buffalo

Analyst:	JFR			**Batch #**	9D10075	**STD 1**		
Date:	4/10/2009					**ACTUAL:**	2120	mg/Kg
EQL:	100 mg/kg		**Solutions:**	**Hexane**		**RANGE:**	625-2950	mg/Kg
				Methanol	N/A	**STD 2**		
				Acetone	N/A	**ACTUAL:**		mg/Kg
				Misc	N/A	**RANGE:**		mg/Kg

Job#	Sample	Pre Wt (g)	Post Wt#1 (g)	Post Wt#2 (g)	Soil (g)	Result (mg/kg)	Dry Weight (%)	Final Result Dry Weight Corrected (mg/kg)
LCS	D059-632	6.4049	6.4129	6.4129	5.109	1565.86	100	1565.86
MBLK	BLANK	6.4381	6.4392	6.4388	5.1351	136.32	100	136.32
RSD102	1	6.434	6.4342	6.4342	5.3048	37.70	3.2	1178.18
	01MD	6.3815	6.3811	6.381	5.0471	-178.32	3.2	-5572.51
LCS	D059-632	6.4387	6.4437	6.4432	5.0718	887.26	100	887.26
MBLK	BLANK	6.4309	6.426	6.4263	5.0418	-912.37	100	-912.37
						#DIV/0!		#DIV/0!
						#DIV/0!		#DIV/0!
						#DIV/0!		#DIV/0!
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Attachment 17.4
Wet Chemistry Batch Cover Sheet

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

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	1st Level	2nd 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 > 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 *immediately* 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:

Data Scanned in _____

WC-Data Review Coversheet
Rev. 1
September 28, 2011

Title: Sample Preparation of Waters for Total, Total Recoverable, or Dissolved Metals for Analysis by ICP-AES methods 6010 or 200.7 [Method No(s). 3005A]

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

E. Scott Wagner Department Manager	10/2/2013 Date	Jennifer Pierce Operations Manager	10/2/2013 Date
Brad Prinzi Quality Assurance Manager	10/2/2013 Date	Christopher Spencer Laboratory Director	10/3/2013 Date

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP is based on Method 3005A – sample preparation of waters for Total, Total Recoverable or Dissolved metals for analysis by ICP-AES.

1.2 The digestion procedure in this SOP is modified. A hot block is used in place of a hot plate for sample heating. The digestion vessels are changed to 50 mL polypropylene digestion cups. Sample size and the amount of chemicals used are also reduced.

1.3 Analytes, Matrix(s), and Reporting Limits

1.3.1 The digestion procedure is used for the preparation of ground waters, surface waters, drinking waters and waste waters for analysis by inductively coupled plasma atomic emission spectroscopy (ICP-AES).

1.3.2 Total Metals, Total Recoverable Metals, and Dissolved (Soluble) Metals: The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is diluted to volume.

1.3.3 Samples prepared by Method 3005A may be analyzed by ICP-AES methods 6010 or 200.7 for the following elements:

Aluminum	Calcium	Magnesium	Sodium
Antimony	Chromium	Manganese	Strontium
Arsenic	Cobalt	Molybdenum	Sulfur
Barium	Copper	Nickel	Thallium
Beryllium	Iron	Potassium	Tin
Boron	Lead	Selenium	Titanium
Cadmium	Lithium	Silicon	Vanadium
		Silver	Zinc

1.3.4 Reporting limits for the above elements are not directly applicable to the digestion procedure alone, and are detailed in the SOP covering analytical methods 6010B and 200.7, see (BF-ME-009).

1.3.5 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:** Total and Total Recoverable Metals: All aqueous samples are acid digested. A 50 mL aliquot of an acidified water sample is digested with 3 mL of concentrated Nitric acid and 2.5 mL of concentrated Hydrochloric acid. The total volume of the sample is reduced to 25 mL by gently heating. After cooling, the digestate is diluted to a final volume of 50 mL with reagent water.

2.1 Dissolved Metals - Aqueous samples to be prepared for Dissolved Metals are filtered prior to initial acidification (preservation), typically at the time of collection. If the laboratory is required to filter and preserve a sample, there is a waiting period of 24hrs before digesting the sample. Otherwise, the digestion procedure is identical to that for Total Metals.

3.0 Definitions

3.1 Total Recoverable Samples – According to SW-846 (Chapter 3), Total Recoverable Metals are metals determined in a sample following the treatment with hot diluted mineral acid.

3.2 Total Metals – The concentration determined on an unfiltered, acidified sample following digestion.

3.3 Soluble Metals – The concentration determined on a filtered, acidified sample following digestion.

4.0 Interferences

4.1 Interferences are discussed in the referring analytical method.

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 this document. 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 Many of the metallic elements analyzed in this method are known to be hazardous to health. Care should be taken in the handling and disposing of all standards and samples.

5.3 Gloves should be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care will be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

5.4 Specific Safety Concerns or Requirements:

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

5.5 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 MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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

6.0 Equipment and Supplies

6.2 *Environmental Express* Hot Block
6.3 *Environmental Express* 50 mL polypropylene digestion cups with caps
6.4 *Eppendorf* pipettes and pipette tips
6.5 Repipettors for dispensing acids
6.6 NIST Certified Thermometer that covers range of 0-150°C.
6.7 Analytical Balance

7.0 Reagents and Standards

7.2 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

.

7.3 Laboratory Reagent Water. Reagent water used for this method is obtained from an ELGA system in the metals analysis laboratory. See SOP BF-GP-002 sections 5.15 – 5.19 for more detailed information concerning Laboratory Reagent Water.

7.4 Trace metal grade concentrated nitric acid (HNO_3) and hydrochloric acid (HCl). Whenever the purity of the acid is suspected, the acid will be analyzed by ICP-MS to determine levels of impurities. If impurity concentrations are at such levels that method blanks are <MDL, the acid can be used.

7.5 Spike standards:

7.5.1 10 μg/mL Ag is prepared by diluting 1.0 mL of 1000 μg/mL Ag stock standard with 2% HNO_3 to 100 mL.
7.5.2 40 μg/mL Sn is prepared by diluting 4.0 mL of 1000 μg/mL Sn stock standard with 2% HNO_3 to 100 mL
7.5.3 2000 μg/mL Si is prepared by diluting 20 mL of 10,000 μg/mL Si stock standard with 2% HNO_3 to 100 mL

7.5.4 ICP-AES spikes:
7.5.4.1 Custom spike solutions: ICUS-3097 (W2) and ICUS-1370 (W1), used directly as purchased. See table 1 for analytes and concentrations.
7.5.4.2 Prepared spike solutions: 10 μg/mL Ag, 40 μg/mL Sn and 2000 μg/mL Si. Prepared as described above.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are to be collected in plastic containers and preserved to a pH of <2 with Nitric Acid. Preserved samples can be stored at room temperature. Sample digestion and analysis must be completed within 180 days of sample collection.

8.2 Samples received at the laboratory unpreserved are kept at 4°C and should be preserved as soon as possible. Allow laboratory preserved samples to sit for 24h prior to digestion.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 Sample QC

9.1.1 Method Blank (MB) is a volume of reagent water processed through each sample preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a Method Blank is carried through the preparation and analysis procedure. This blank is useful in monitoring any contamination.

9.1.2 Laboratory Control Sample (LCS) is a volume of reagent water spiked with known concentrations of analytes and carried through the preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), an LCS must be employed to determine method accuracy.

9.1.3 Matrix Spikes: For each batch of samples (not to exceed 20 samples), a matrix spike (MS) should be processed on a routine basis. Replicate samples will be used to determine matrix effects on digestion and detection.

9.1.4 Duplicates: For each batch of samples (not to exceed 20 samples), replicate samples are to be processed on a routine basis. Replicate

samples are either a method duplicate (DU) or matrix spike duplicate (MSD) depending on the clients' request, but are usually spike duplicates. Replicate samples will be used to determine precision. An DU is another aliquot of the selected sample. An MSD is another MS that is processed through the preparation and analysis procedure.

9.2 Data Assessment and Acceptance Criteria for Quality Control Measures

A representative digestion batch and the quality control criteria is illustrated below

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Reporting Limit (RL)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	80-120% Recovery (SW846); 85-115% Recovery (MCAWW)
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125% Recovery
Matrix Spike Duplicate (MSD)[1]	1 in 20 or fewer samples	75-125% Recovery; Relative % Difference <20%

[1] The sample for MS/MSD is randomly selected, unless specifically requested by a client.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Method Blank (MB): For each digestion batch of 20 samples or less, transfer 50 mL of laboratory reagent water to a digestion cup and carry through the entire analytical process.

10.1.2 Laboratory Control Sample (LCS): For each digestion batch of 20 samples or less, transfer 50 mL of laboratory reagent water to a digestion cup and carry through the entire analytical process. Add the appropriate amount of spikes according to the determination methods:

10.1.2.1 ICP-AES: Fortify with 0.25 mL each of ICUS-3097 (W2), ICUS-1370 (W1), 10 μg/mL Ag, 40 μg/mL Sn and 2000 μg/mL Si spiking solutions.

10.1.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify two aliquots with the same spikes and amounts as listed above for the LCS. These three samples are the base (source) sample, MS and MSD.

10.1.4 Matrix Duplicate (DU) and Matrix Spike (MS): For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify one aliquot with appropriate amount of spikes as listed in 10.1.2.1. These three samples are the base (source) sample, DU and MS. Note: The DU/MS combination is an alternative to the MS/MSD combination and is not routinely prepared for this method. It may be done on the basis of the clients' requests.

10.1.5 Samples: Homogenize each sample as described in SOP BF-GP-005 section 5.5. Pour 50 mL of sample into a clean, labeled digestion cup. Add 3 mL conc. HNO_3 and 2.5 mL conc. HCl to each cup. Heat samples to a temperature of 95±3 °C on a hot block. Evaporate samples down to 25mL (approximately 3-3.5 hours). Remove from hot block and allow samples to cool. Bring final volume to 50 mL with reagent water and cap.

10.1.6 Turbid samples may be filtered with 2 µm Teflon filters prior to analysis.

10.2 Calibration and Standardization

10.2.1 Class A volumetric glassware is to be used for the preparation of spiking solutions.

10.2.2 The Environmental Express digestion cups are Class-A calibrated. Certificates are kept in the digestion lab.

10.2.3 Hot block temperatures are to be checked daily and entered into the digestion block log. The temperature of 25-50 ml of water in digestion is recorded for a representative sample location on the hot block. (The location is selected on a rotating basis.) This is done using a NIST verified thermometer.

10.2.4 Pipettes/Eppendorf's are calibrated quarterly and verified daily by a delivery of reagent water on a Certified Balance. Results are adjusted for temperature. (See SOP BF-GP-001.)

10.2.5 Analytical Balance is to be verified daily using certified Class 1 weights. (See SOP BF-GP-002.)

10.2.6 Thermometers are to be calibrated against a NIST certified thermometer as described in SOP BF-GP-020. Thermometers are to be tagged with the correction factor and calibration due date.

11.0 Calculations / Data Reduction

11.1 Calculations: Applicable calculations are covered under the corresponding analysis method SOP.

11.2 Contingencies for Handling Out-of-Control or Unacceptable Data

11.2.1 Contingencies for unacceptable data will have to be evaluated on a client-by-client or even by a sample-by-sample basis by the supervisor, the lab director or the project manager. Corrective action will be prescribed accordingly.

11.2.3 Batches with unacceptable quality control results may need to be reanalyzed and/or re-digested for the affected analytes.

11.2.4 A job exception form should be completed for the following issues

- Insufficient sample for digestion (<50 mL)
- Unusual Matrix / Matrix Reactivity
- Loss of Digestate
- Holding Time exceedence

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present.

12.1.2 The MDLV study is conducted annually in accordance with SOP BF-QA-001

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 MSDS 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 perfomed 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 Method 3005A from: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW846, Third Edition and updates.

15.2 The following SOPs are referenced as being supplemental to the material presented in this SOP:

15.2.1 BF-ME-009 – Corresponding method SOP (6010B) used for the analysis of digests generated by this and other methods.

15.2.2 BF-GP-001 – Pippette/Eppendorf Calibration

15.2.3 BF-GP-002 – Laboratory Reagent Water; Balance Calibration

15.2.4 BF-GP-020 – Thermometer Calibration

15.2.5 BF-QA-001 – Method Detection Limits

15.2.6 BF-QA-004 – Training and DOCs

15.2.7 BF-WM-001 – Waste Management

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16.0 Method Modifications:

16.1 A hot block is used in place of a hot plate for sample heating.
16.2 The digestion vessels are changed to 50 mL polypropylene digestion cups.
16.3 Sample size and the amount of chemicals used are also reduced.
16.4 Adopted prep method 3005 for 200.7 water samples. A volume of 50 mL initial and 50 mL final is used instead of 100 mL, 3 mL concentrated nitric and 2.5 mL concentrated HCl instead of 2 mL (1+1) Nitric and 1 mL (1+1) HCl. Utilize hot block instead of hot plate set at 95 degrees Celsius instead of 85 degrees Celsius. No

watch glass for digestion. There are hot blocks in individual fume hoods to prevent contamination. No reflux performed on the samples. Samples that may clog nebulizer are filtered with 2.0 micron filters. Digest down to <25 mL (approximately 3 hours).

17.0 Attachments

17.1 TABLE 1: ICP-AES Spikes
17.2 Sample Digestion Log

18.0 Revision History

- Revision 3, dated November 4, 2013
  - Updated section 10.1.4: corrected referenced section from 12.2.1 to 10.1.2.1 to show spikes added and concentrations used for matrix spikes.
  - Changed section 10.2.4 eppendorf calibration from monthly to quarterly as specified in SOP BF-GP-001 (General Practices).
  - Changed lot identification of W2 standard from ICUS-574 to ICUS-3097 (currently in use) in sections 7.5.4.1 and 10.1.2.1.
  - Updated Table1 to include sodium spiking concentration for ICUS-3097 and Molybdenum final concentration.
  - Updated attachments 17.2 (Sample Digestion Log).
  - Corrected section 12.1.2 to reference MDLV studies are performed annually.
- Revision 2, dated September 12, 2011
  - Added new elements to section 1.3.3
  - Added silicon spike information to sections 7.4 and 10.1.2.1
  - Section 9.0 and 10.0. Corrected LIMS name abbreviations for batch quality control samples
  - Added Section 16.4 to Method Modifications Section (Adoption of Prep Method 3005 for 200.7 samples).

- Updated TABLE 1.
- Updated Sample Digestion Log.
- Quality Manager change, signature added.

o Revision 1, dated August 25, 2009

- Integration and updated attachments for new LIMs system
- Department Supervisor change, signature updated.

Analyte	ICUS-1370 (ug/ml)	ICUS-3097 (ug/ml)	10 ug/ml Ag (ug/ml)	40ug/ml Sn (ug/ml)	2000 ug/ml Si (ug/ml)	Final (ug/ml)
Aluminum		2000				10
Antimony	40					0.2
Arsenic	40					0.2
Barium		40				0.2
Beryllium	40					0.2
Boron		40				0.2
Cadmium	40					0.2
Calcium	2000					10
Chromium	40					0.2
Cobalt	40					0.2
Copper	40					0.2
Iron	2000					10
Lead	40					0.2
Lithium		40				0.2
Magnesium	2000					10
Manganese	40					0.2
Molybdenum	40					0.2
Nickel	40					0.2
Potassium		2000				10
Selenium	40					0.2
Silicon					2000	10
Silver			10			.05
Sodium		2000				10
Strontium		40				0.2
Thallium	40					0.2
Tin				40		0.2
Vanadium	40					0.2
Zinc	40					0.2
Titanium	40					0.2

17.1 Table 1 (above) ICP-AES Spike Analytes and Concentrations

17.2 Sample Digestion Log (1 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101580
Method Code: 480-3005A_TOT-480

Analyst: Mantell, Jaclyn

Batch Open: 1/31/2013 7:55:00AM
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~480-101580/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		MB 480-101580/1-A
2	LCS~480-101580/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		LCS 480-101580/2-A
3	480-32215-I-1 (200.7)	N/A	Water	50 mL	50 mL	1/31/13	1_Day_RUSH -	2		480-32215-1-1-A
4	480-32215-I-2 (200.7)	N/A	Water	50 mL	50 mL	1/31/13	1_Day_RUSH -	2		480-32215-1-2-A
5	480-32215-I-2~MS (200.7)	N/A	Water	50 mL	50 mL	1/31/13	1_Day_RUSH -	2		480-32215-1-2-B MS
6	480-32215-I-2~MSD (200.7)	N/A	Water	50 mL	50 mL	1/31/13	1_Day_RUSH -	2		480-32215-1-2-C MSD

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17.2 Sample Digestion Log Continued (2 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101580
Method Code: 480-3005A_TOT-480

Analyst: Mantell, Jaclyn

Batch Open: 1/31/2013 7:55:00AM
Batch End:

	Input Sample Lab ID (Analytical Method)	(Sub-List)	Analytes
1	MB 480-101580/1 N/A	N/A	N/A
2	LCS 480-101580/2 N/A	N/A	N/A
3	480-32215-I-1 (200.7)	(Local Method)	Al, Cr, Cu, Fe, Mn, Ni, Sb, Zn
4	480-32215-I-2 (200.7)	(Local Method)	Al, Cr, Cu, Fe, Mn, Ni, Sb, Zn
5	480-32215-I-2 MS (200.7)	N/A	N/A
6	480-32215-I-2 MSD (200.7)	N/A	N/A

Analytes that are not being reported with be displayed in [...] brackets. Analytes that are not being reported but are on the spike list with be displayed in (...) parentheses.

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17.2 Sample Digestion Log Continued (3 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101580 Analyst: Mantell, Jaclyn Batch Open: 1/31/2013 7:55:00AM

Method Code: 480-3005A_TOT-480 Batch End:

Batch Notes

Digestion Tube/Cup Lot #	1208057
Hot Block ID number	B
Hood ID or number	
Nitric Acid Reagent ID Number	0000021587
Hydrochloric Acid Reagent ID Number	4112030
Uncorrected Temperature	95.0
Oven, Bath or Block Temperature 1	95.5
Uncorrected Temperature 2	97.2
Oven, Bath or Block Temperature 2	97.7
ID number of the thermometer	111404326
Filter Paper Lot Number	
Pipette ID	MDL-3
First Start time	755
First End time	1055
Batch Comment	

Comments

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17.2 Sample Digestion Log Continued (4 of 5)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101580
Method Code: 480-3005A_TOT-480

Analyst: Mantell, Jaclyn

Batch Open: 1/31/2013 7:55:00AM
Batch End:

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 480-101580/2	MED_01_Si_00019	0.25 mL	50 mL		
LCS 480-101580/2	MED_01_W1_00011	0.25 mL	50 mL		
LCS 480-101580/2	MED_02_W2_00011	0.25 mL	50 mL		
LCS 480-101580/2	MED_03_Ag_00028	0.25 mL	50 mL		
LCS 480-101580/2	MED_04_Sn_00025	0.25 mL	50 mL		
480-32215-I-2 MS	MED_01_Si_00019	0.25 mL	50 mL		
480-32215-I-2 MS	MED_01_W1_00011	0.25 mL	50 mL		
480-32215-I-2 MS	MED_02_W2_00011	0.25 mL	50 mL		
480-32215-I-2 MS	MED_03_Ag_00028	0.25 mL	50 mL		
480-32215-I-2 MS	MED_04_Sn_00025	0.25 mL	50 mL		
480-32215-I-2 MSD	MED_01_Si_00019	0.25 mL	50 mL		
480-32215-I-2 MSD	MED_01_W1_00011	0.25 mL	50 mL		
480-32215-I-2 MSD	MED_02_W2_00011	0.25 mL	50 mL		
480-32215-I-2 MSD	MED_03_Ag_00028	0.25 mL	50 mL		
480-32215-I-2 MSD	MED_04_Sn_00025	0.25 mL	50 mL		

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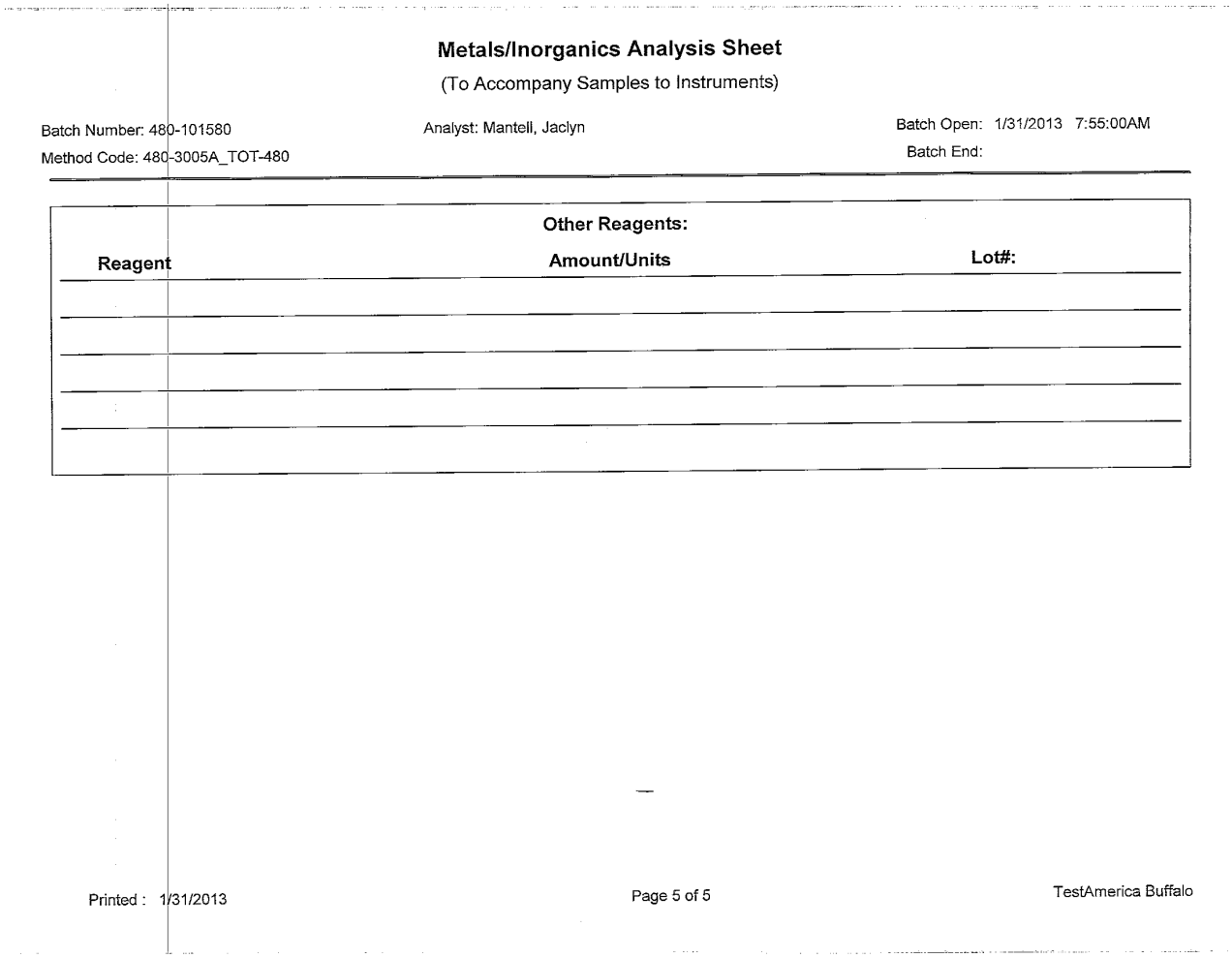
17.2 Sample Digestion Log Continued (5 of 5)



Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101580
Method Code: 480-3005A_TOT-480

Analyst: Mantell, Jaclyn

Batch Open: 1/31/2013 7:55:00AM
Batch End:

Other Reagents:

Reagent	Amount/Units	Lot#:

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TestAmerica Buffalo

Title: **METHOD 3050B: ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS**

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Approvals (Signature/Date):

E. Scott Wagner 2/24/14 E. Scott Wagner Date Department Manager	Kenneth Kasperek 2/24/14 Kenneth Kasperek Date Health & safety Manager
Brad Prinzi 2/24/14 Brad Prinzi Date Quality Assurance Manager	Chris Spencer 2/24/14 Chris Spencer Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This method is an acid digestion procedure used to prepare sediment, oil, waste, sludge, and soil samples for analysis by inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma-mass spectroscopy (ICP-MS). This method may be digested using The Environmental Express Auto Block or by manually adding reagents and heating on hot blocks.

1.1 Analytes, Matrix(s), and Reporting Limits

Samples prepared by this method may be analyzed for the following metals in soils, sludges, or sediments. However, not all analytes are analyzed by each method.

Aluminum	Cobalt	Silver	Strontium
Antimony	Copper	Sodium	
Arsenic	Iron	Thallium	
Barium	Lead	Selenium	
Beryllium	Magnesium	Titanium	
Boron	Manganese	Vanadium	
Calcium	Molybdenum	Zinc	
Cadmium	Nickel	Tin	
Chromium	Potassium	Lithium	

See SOP BF-ME-009 Section 22.3 for soil detection limits for all analytes listed above.

2.0 Summary of Method

A representative sample (0.5 g ± 0.05g - wet weight) is digested in Nitric acid and Hydrogen Peroxide. The digestate is then refluxed with Hydrochloric acid as the final reflux. All digestates are diluted to a final volume of 50 ml with laboratory reagent water. A separate sample aliquot shall be dried for a total solids determination.

3.0 Definitions

3.1 Total Metals --The concentration determined on filtered sample following digestion. Note that this method is designed to determine total *environmentally available* metals.

4.0 Interferences

4.1 Sludge samples can contain diverse matrix types, each of which may present an analytical challenge. Spiked samples and any relevant standard reference material are processed in accordance with the quality control requirements to aid in determining whether this method is applicable to a given waste.

4.2 Boron from glassware will leach into the sample solution during and following sample processing. For critical low level determinations of Boron, only quartz and/or plastic labware is used.

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

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

5.1.1 Laboratory coats, gloves and safety glasses shall be worn during all stages of the digestion procedure.

5.1.2 Nitrile gloves shall be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care must be taken when dispensing concentrated acids. Concentrated acids must be dispensed only in the fume hood.

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

5.1.4 Hydrogen peroxide (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 to avoid a reaction and possible violent effervescence, or boiling over of the digestate.

5.1.5 Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care must be taken in the handling and disposing of all standards and samples. See section 20.0 for procedures on the disposal of standard and sample waste.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
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.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
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

- *Environmental Express* Auto Block/with PDA and Digestion Blocks
- 50 ml Polypropylene digestion cups with reflux caps from Environmental Express
- Sample racks-polycarbonate
- Filter Mate Teflon press filter
- Membrane disk filters 0.45 um 47mm Pall Life Sciences
- Eppendorf pipettes and pipette tips
- Analytical balance capable of accurate weighing to 0.01 g.
- Drying oven maintained at 105±4°C.
- NIST Certified Thermometer - That covers range of 0-150°C.
- Silicon (IV) Oxide beads
- **Maintenance Supplies for the Autoblock from Environmental Express**:
- The PDA will issue a maintenance indicator message informing analyst to schedule this maintenance:
- HEPA Filter Replacement---every 12 months
- Peristaltic Pump Tube---Flexible tubing-every 12 months
- Reagent Injection Kit---Tubing Assembly from Splitter to Sample Cup,6 lines-every 12 months

- Reagent Uptake Line---25 feet of reagent uptake line for 5 bottle-to-inlet lines-as required

6.1 Instrumentation

Environmental Express Auto-Block w/PDA

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents confirm the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Laboratory Reagent Water. See SOP No. BF-GP-002

7.3 Trace metal grade concentrated nitric acid (HNO_3) and hydrochloric acid (HCl). The certificate of analysis is directly on the acid bottle. Whenever the purity of the acid is suspected, the acid is analyzed via ICPMS to determine levels of impurities. If impurity concentrations are at such levels that method blanks are <MDL, the acid can be used.

7.4 1:1 HNO_3, is prepared by mixing equal volume of reagent water and concentrated nitric acid. Pour concentrated acid to water; never pour water to concentrated acid.

7.5 30% Hydrogen Peroxide, H_2O_2, un-stabilized 30% Hydrogen Peroxide used if analysis requires Tin.

7.6 Spike standards:

7.6.1 10 μg/ml of Ag is prepared by filling a class "A" volumetric flask half way with laboratory reagent water and adding four mls of Nitric Acid. Pipette 1.0 ml of 1000 μg/ml Ag stock standard to the 100 ml volumetric flask and fill to the line with laboratory reagent water. Spike standard to be verified via ICP-AES prior to use.

7.6.2 40 μg/ml of Sn is prepared by filling a class A volumetric flask half way with laboratory reagent water and adding four mls of Nitric Acid. Pipette 4.0 ml of 1000 μg/ml Sn stock standard to a 100 ml volumetric flask and fill to the line with laboratory reagent water. Spike standard to be verified via ICP-AES prior to use.

7.6.3 ICP-AES spikes: ICUS-1370, ICUS-574, 10 μg/mL Ag and 40 μg/mL Sn. See Table 1 for details.

7.6.4 ICP-MS spikes: See Table 1.

7.6.5 The SRM sample is purchased from Environmental Resource Associates. The concentration of each analyte is different from lot to lot. The certified concentrations are stored in the binder with the Certificates of Analysis.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	poly	300 mls	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soil	Glass	3 grams	Cool 4 $\pm$ 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 For each batch of samples (not to exceed 20 samples), a preparation blank (method blank, BLK) is carried throughout the entire sample preparation and analytical process. This blank is useful in monitoring any contamination.

9.2 For each batch of samples (not to exceed 20 samples), a Standard Reference Material (SRM) must be employed to demonstrate proper implementation of the method.

9.3 For each batch of samples (not to exceed 20 samples), a matrix spike (MS) is processed on a routine basis. The MS will be used to determine matrix effects on digestion and detection.

9.4 For each batch of samples (not to exceed 20 samples), a replicate sample is processed on a routine basis. Replicate samples are either method duplicates (DUP) or spike duplicates (MSD) depending on the clients' request, but are usually spike duplicates. Replicate samples will be used to determine precision. DUP is another aliquot of the selected sample. MSD is another MS that is processed through the preparation and analysis procedure.

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit or < 2.2MDL for MCAWW
Standard Reference material (LCSSRM)	1 in 20 or fewer samples	Specified by manufacturer of SRM
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
MS Duplicate (MSD)[1]	1 in 20 or fewer samples	75-125%
Matrix Duplicate (DU)[1]	1 in 20 depending on method/client request	20%

[1] The sample selection for MS/MSD/DU is determined by the extraction lab unless specifically requested by a client.

10.0 Procedure

10.1 Method Blank (MB): For each digestion batch of 20 samples or less, weigh 0.5 ± 0.05 grams of Silicon Oxide to a digestion cup. Record the weight on the batch sheet. The Silicon Oxide is used as a soil substitute. Next, transfer 10 mls of laboratory reagent water to the digestion cup and carry it through the entire analytical process.

10.2 Laboratory Control Sample Standard Reference material (LCSSRM): For each digestion batch of 20 samples or less, weigh 0.5 ± 0.01grams of LCS to a digestion cup and carry it through the entire digestion process.

10.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify two aliquots with appropriate amount of spikes according to the determination methods. These three samples are 'Sample', 'MS', and 'MSD'.

ICP-AES and ICP-MS: Fortify with 0.50 mL ICUS-1370, 0.50 ml ICUS-3097, 0.50 mL 10 μg/ml Ag, and 0.50 mL 40 μg/ml Sn spiking solutions. (Table 1)

10.4 Matrix Duplicate: For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify one of the aliquots with appropriate amount of spikes according to the determination methods described in 10.03. These three samples are treated as 'Sample', 'DU' and 'MS'. A matrix duplicate is not routinely prepared. It is only done on the basis of the clients' requests.

10.5.0 Sample Digestion via manual method

10.5.1 Mix the sample thoroughly to achieve homogeneity. Weigh out 0.5 grams of sample ±0.05 grams to a 50ml digestion cup.

10.5.2 Weigh out 0.5 grams of SRM ±0.001 grams to a digestion cup.

10.5.3 Add 10.0mls of laboratory reagent water to each digestion cup

10.5.4 Spike the Matrix Spike and Matrix Spike Duplicate as stated in 10.3

10.5.5 Add 5.0mls of 1:1 Nitric Acid and heat for 15 minutes with a reflux cap.

10.5.6 Cool for 10 minutes

10.5.7 Add 2.5mls of concentrated Nitric Acid and heat for 30 minutes with a reflux cap.

10.5.8 Cool for 5 minutes.

10.5.9 Add 3.0 mls of reagent water, wait 1 minute, and add 1.0 ml of Hydrogen Peroxide.

10.5.10 Wait 5 minutes.

10.5.11 Add 1.0ml of Hydrogen Peroxide.

10.5.12 Heat for 8 minutes with a reflux cap.

10.5.13 Cool for 5 minutes.

10.5.14 Add 1.0 ml of Hydrogen Peroxide

10.5.15 Wait 3 minutes

10.5.16 Add 2.0 mLs of Hydrogen Peroxide

10.5.17 Wait 3 minutes.

10.5.18 Heat for 8 minutes.

10.5.19 Cool for 5 minutes.

10.5.20 Add 3.0mls of reagent water and 5.0mls of concentrated Hydrochloric Acid.

10.5.21 Heat for 15 minutes with a reflux cap.

10.5.22 Allow sample to cool. Wash down the digestion cup walls and reflux cap with reagent water. Bring the final volume to 50mls with reagent water.

10.5.23 Filter each sample with a filter.

10.6 Sample digestion via Auto-block

10.6.1 Verify that temperature is set to 115°C in the temperature control block.

10.6.2 Prepare samples as instructed under the manual method sections 10.5.1 through 10.5.4.

10.6.3 Place samples in auto-block. Make sure that the last row is completely filled or reagents will be added to empty block tubes.

10.6.4 Tap the “Select Mode” button in the Manual Mode window.

10.6.5 Tap the “Select Mode” button in the Service Mode window.

10.6.6 Verify that the method selected is 3050_Modified_6-17-11.ROM.

10.6.7 Tap the “Start” button

10.6.8 At the cannot Verify Reagent H2O2 message, tap “OK”

10.6.9 Verify that all reagents have adequate volume, then tap “OK”

10.6.10 Select the correct number of rows for the run, and then tap the “Apply” button.

10.7 Calibration

10.7.1 Environmental Express digestion cups arrive with a volume certification these certificates are kept in the digestion lab. Each lot of cups is verified at the 50mL mark and recorded.

10.7.2 Analytical balances are checked and calibrated using NIST Class "1" Certified weights (See SOP BF-GP-002) daily and entered into a logbook.

10.7.3 Hot block/Auto-Block temperatures are to be checked daily and entered into the digestion log. This is done using a NIST certified thermometer. Acceptance range is 95 degrees ±3 degrees.

10.7.4 Pipettes/ Eppendorf's are verified daily and calibrated every 3 months by a delivery of reagent water on a Certified Balance (See SOP BF-GP-001). This information is logged into a spreadsheet.

10.7.5 Auto-block Reagent pump calibration: all 6 lines are calibrated quarterly to insure accuracy of volumes injected. Per manufacturer ±2% at 10mls is acceptable. These are logged in a spreadsheet.

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10.7 Sample Analysis

Refer to Analytical Sop’s BF-ME-009 and BF-ME-010.

11.0 Calculations / Data Reduction

11.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

11.2 If percent solid is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample. See SOP #BF-GP-004.

11.3 Accuracy

$$\textbf{ICV / CCV, SRM \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.4 Precision (RPD)

Matrix Duplicate (DU) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.5 Concentration = 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.

11.6 Method Performance On an annual basis, Method Detection Limit studies are performed in accordance with 40 CFR 136, Appendix B.

11.7 Demonstration of Capabilities

Doc's are performed on each method from each analyst once a year and approved by the laboratory manager as well as the Quality Manager.

11.8 Training Requirements

The QA Manual or the SOP will be referenced for training requirements.

12.0 Pollution Control

All samples, reagents, and laboratory wastes must be handled with caution. Appropriate safety measures should be employed as detailed in TestAmerica's Laboratory Safety Manual and Chemical Hygiene Plan. All waste will be disposed of in accordance with Federal, State and Local regulations. Where it is 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."

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).

13.0 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 TestAmerica's Safety Manual.

14.0 References / Cross-References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW846, Third Edition; Final Update II; September 1994.

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15.0 Method Modifications:

Item	Method	Modification
1) Volume	3050B	Initial Wt 0.5 g: Final Vol. 50mls (All Acid volumes are adjusted accordingly)

16.0 Attachments:

Attachment 1: Spike concentrations
Attachment 2: Digestion Batch sheet

17.0 Revision History

- Revision 3, dated February 24, 2014
  - QA Manager Updated, signature added

- Revision 2, dated January 13, 2012
  - Replaced SRM with LCSSRM. Throughout.
  - Replaced BLK with MB. Throughout.
  - Replaced DUP with DU. Throughout.
  - Edited sample amounts from 0.5g to 0.5 ± 0.05 grams.
  - Edited LCSSRM amounts from 0.5g to 0.5 ± 0.01 grams
  - Section10.5.20 Replaced 2.5 mL of concentrated Hydrochloric Acid to 5mL.
  - Section 10.6.6 changed method file from 3050_COOLINGTEST.ROM to 3050_Modified_6-17-11.ROM.
  - Changed Quality Manager, signature added.

- Revision 1, dated February 8, 2010
  - Updated for Element
  - Auto-block method separated and updated

- Revision 0, dated January 25, 2008
  - Integration for TestAmerica and STL operations.
  - Section 6.0: Corrected drying oven temperature range
  - Section 10.03: Deleted ICPMS spike
  - Section 10.1.1: Corrected from 0.2 g to 0.5 g
  - Table 2: Deleted

Attachment 1

(Table 1) Soil spikes

Analyte	ICUS-1370 (µg/mL)	ICUS-574 (µg/mL)	10 µg/Ml Ag Stock (µg/mL)	40 µg/mL Sn Stock (µg/mL)	Final Conc. In Digestate (ug/mL)	Final Conc. In Soil Sample (mg/kg)
Aluminum		2000			10	1
Antimony	40				.2	.02
Arsenic	40				.2	.02
Barium		40			.2	.02
Beryllium	40				.2	.02
Boron		40			.2	.02
Cadmium	40				.2	.02
Calcium	2000				10	1
Chromium	40				.2	.02
Cobalt	40				.2	.02
Copper	40				.2	.02
Iron	2000				10	1
Lead	40				.2	.02
Magnesium	2000				10	1
Manganese	40				.2	.02
Molybdenum	40				.2	.02
Nickel	40				.2	.02
Potassium		2000			10	1
Selenium	40				.2	.02
Silver			10		.05	.005
Sodium		2000			10	1
Thallium	40				.2	.02
Tin				40	.2	..02
Vanadium	40				.2	.02
Zinc	40				.2	.02
Titanium	40				.2	.02

Attachment 2: Digestion Batch sheet

PREPARATION BENCH SHEET

9L31004

TestAmerica Buffalo

Matrix: Solid **Prepared using: Metals - 3050B** **Printed: 1/5/2010 3:10:32PM**

Lab Number	Prepared	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Comments

Eppendorfs used: MDL-4
Hot Block Temp: 115/F
Sample Temp: 94
Digestive cup lot: A905LS269
2 micron Filtermate:

Reagent
9070334 DIG SiO2
9101250 DIG Hydrochloric Acid soil
9110350 DIG Hydrogen Peroxide, stabilized
9120043 DIG Nitric Acid soil

L2

Criteria:

Initial Calibration/Second Source Criteria Met?	Y	N
CCV/CCB Criteria Met?	Y	N
Method Blank Criteria Met?	Y	N
LCS Criteria Met?	Y	N
MS/SD Criteria Met?	Y	N
ICSA ICSAB LCV Criteria Met?	Y	N

Dig. Analyst: _____ Date: _____
Analyst: _____ Date: _____
Entry: _____ Date: _____
Review: _____ Date: _____

Comments: _____

Work Orders

RSL1012

RSL1045

RSL1102

Page 3 of 3

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION PROCEDURE - USING REDUCED VOLUME (METHOD No. 3510C RVE/LVI)

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

Michelle Freeman	2/15/13 Date	Kenneth Kasperek	2/12/13 Date
Department Manager		Technical Director	
Brad Prinzi	2/12/13 Date	Christopher Spencer	2/12/13 Date
Quality Assurance Manager		Laboratory Director	

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This method is used to extract a broad range of organic compounds from aqueous samples for analysis by either GC or GCMS. This method also describes concentration techniques, which prepare the extract for the appropriate analysis.

1.1 Analytes, Matrix(s), and Reporting Limits

1.2 Analytes: water-insoluble and slightly water-soluble organics

1.3 Matrices: aqueous samples (water)

1.4 Reporting limit: N/A

1.5 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 A measured volume of aqueous sample, approximately 250 mL, is extracted with methylene chloride at a specified pH using separatory funnel extraction. The extract is dried through activated anhydrous sodium sulfate, concentrated using a Nitrogen blowdown technique, and if necessary solvent exchanged into a solvent suitable for its cleanup or analysis.

3.0 Definitions

3.1 Standard definitions are found in Section 3.0 of the Laboratory Quality Manual.

3.2 Solvent exchange: The process of exchanging the solvent of the sample extract from the extraction solvent (usually methylene chloride) to the final method solvent (usually hexane).

4.0 Interferences

4.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

4.2 Matrix interference may be caused by contaminants that are co-extracted from the sample.

4.3 Glassware used for water extractions is kept separate from soil glassware to prevent cross-contamination.

4.4 Basic extraction conditions can cause the decomposition of some analytes including: organochlorine pesticides, phthalate esters and phenols

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

5.1.1 The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples.

5.1.2 All parameters of this extraction must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

5.1.3 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.4 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

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
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.
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 Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
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.
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

6.1 250 mL graduated cylinders
6.2 100 mL graduated cylinders
6.3 Teflon separatory funnels, stopcocks, and caps
6.4 Syringes
6.5 Turbo Vaps equipped with Nitrogen blowdown
6.6 Turbo Vap vessels calibrated @ 1.0mL
6.7 16 oz. French squares
6.8 Steel funnels
6.9 Glass wool
6.10 Disposable pipettes and bulbs
6.11 2 mL vials and caps (amber or clear depending on application)
6.12 4 mL glass vials with caps and (PTFE)- lined cap inserts
6.13 Vial crimpers
6.14 Wide range pH paper
6.15 Centrifuge and centrifuge tubes
6.16 Automatic separatory funnel rotators
6.17 Narrow range pH paper
6.18 Aluminum weigh dishes

7.0 Reagents and Standards

7.1 **Note:** All solvents are pesticide grade or equivalent
7.2 Methylene chloride delivered in cycletainers
7.3 Hexane delivered in cycletainers
7.4 Acetone delivered in cycletainers
7.5 Methanol

7.6 10N sodium hydroxide
7.7 1:1 sulfuric acid
7.8 Concentrated Sulfuric Acid
7.9 Anhydrous granular sodium sulfate. **Note:** Sodium sulfate must be baked in a 400°C oven for a minimum of 4 hours before use, or alternately may be purchased pre-baked from Jost chemical.
7.10 Deionized water and/or carbon filtered (volatile free) water
7.11 Surrogate and spike solutions appropriate to the final determinative procedures as assigned by test profile.

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.

Test	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
8270	250mL or 1-Liter glass amber w/ Teflon lid	250 mL	Cool to 4˚C	7 days	SW846
8081	250mL or 1-Liter glass amber w/ Teflon lid	250 mL	Cool to 4˚C	7 days	SW846
8082	250mL or 1-Liter glass amber w/ Teflon lid	250 mL	Cool to 4˚C	7 or 365 days	SW846
310.13	250mL or 1-Liter Glass amber	250 mL	None Required- samples may be preserved with sulfuric acid	7 days	NYSDOH
8015	250mL or 1-Liter Glass amber	250 mL	None Required- samples may be preserved with sulfuric acid	7 days	NYSDOH

8.1 Typical method holding time for water samples is seven days from sampling. However, the client may impose a more strict time constraint.

8.2 Clients may request CLP QC requirements without Continuous Liquid/Liquid extraction technique.

9.0 Quality Control

The following quality control samples are prepared with each batch of samples. All method blanks, laboratory control standards, matrix spikes and matrix spike duplicates will undergo the same procedure as the samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Standard (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] Laboratory Control Duplicate Standard (LCSD) is performed only when insufficient sample is available for the MS/SD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client or predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS/LCSD, MS/MSD)
[4] Statistical control limits are performed annually and are updated into LIMS.

RCP/MCP Requirements: A LCS/LCSD must be prepared with every prep batch along with any site specific MS/MSDs. The LCS/LCSD/MS/MSD spiking standard must contain all target analytes at a concentration near the midpoint of the calibration curve being used for analysis.

10.0 Procedure

10.1 Assemble a pre-rinsed 2L separatory funnel, stopcock and stopper as well as all other extraction supplies and glassware. Refer to SOP BF-GP-003 for glassware cleaning instructions.

10.2 Label each separatory funnel with a batch label that corresponds with the batch sample I.D.

10.3 Make a powder funnel by placing a glass wool plug in a steel funnel, and fill the funnel 2/3 full with baked granular sodium sulfate. (For better analyte recoveries, the sodium sulfate can be rinsed prior to extraction with 10-20mL of methylene chloride. Discard this methylene chloride rinse.) Place the powder funnel into a clean Turbovap vessel labeled with batch sample ID labels. (If humidity is a concern, push the end of the powder funnel through a paper napkin to catch any water that may condense on the sides of the funnel during the extraction procedure.)

10.4 Obtain the designated spike and surrogate solutions for the analytical method and allow them to come to room temperature.

10.5 Using wide range pH paper, test the initial pH of the sample and record the pH in the LIMs benchsheet.

10.6 Visually inspect samples for oil layers and sediment. Any sample that contains an oil layer should be brought to the department manager's attention prior to extraction. The department manager will discuss the analysis of the sample with the project manager. The project manager may decide to have both the aqueous and oil layers analyzed separately.

10.7 Samples may be received in 250mL or 1 Liter bottles. The initial volume to be used for the method is 250mL.

10.7.1 **For any sample received in a 1 liter bottle**, measure 250mL of sample into a pre-rinsed graduated cylinder and record this initial volume in the LIMs bench sheet. Discard the remaining sample volume into "A" waste. Proceed to step 10.7.4.

10.7.2 **For samples received in 250 mL bottles**, if the sample is relatively free of sediment, mark the meniscus on the bottle. Once the sample is transferred to the corresponding sep funnel, the empty sample bottle is filled to the meniscus mark with tap water. The tap water is then transferred to a graduated cylinder, and the volume is recorded in the bench sheet. Alternately, the full bottle can be weighed and the volume automatically uploaded into the gross weight section off the LIMs bench sheet. After emptying the volume into the separatory funnel, weigh the empty bottle and record the weight in the tare section of the bench sheet. The difference between the gross weight and tare weight is calculated in the LIMs bench sheet and the difference is the initial volume amount.

10.7.3 **For samples received in 250 mL bottles** that contain a large amount of sediment, the sample volume should be measured by pouring the sample into a pre-rinsed graduated cylinder, leaving as much of the sediment in the sample bottle as possible. Record the initial sample volume amount in the LIMs bench sheet and transfer the sample into the corresponding labeled separatory funnel. Add the appropriate NCM that the sample was decanted.

10.7.4 Record any comments about the samples in the comments section of the bench sheet.

10.8 Measure 250 mL of the appropriate extraction water (see Table 3) for all QC samples (MB, LCS and LCSD) and add to the labeled separatory funnels. All batch QC will undergo the same procedure as batch samples.

10.8.1 Add the appropriate spike to the QC samples (LCS/LCSD/MS/MSD) and add surrogate solutions to all samples.

10.8.2 Samples that have been transferred already to their separatory funnels have the surrogate added directly to the separatory funnel.

10.8.3 Samples remaining in their sample bottles have the surrogate added directly to the sample bottle, recapped and shaken.

10.8.4 It is important to mark the labels of each sample and blank accordingly when adding spikes and surrogates to avoid error. Once a surrogate has been added (whether it is to the original sample jar or the separatory funnel), an "X" must be drawn on the label affixed to the separatory funnel. After a spike has been added to QC samples, circle the "X" on the label to indicate that the sample has received the spike.

10.9 Transfer all samples to their corresponding labeled separatory funnels.

10.9.1 Verify that samples are in the correct method pH range using narrow range pH paper.

10.9.2 Make adjustments to sample pH as needed using 1:1 H_2SO_4 or 10N NaOH. If pH adjustment is required, the separatory funnel must be capped and shaken for a moment to ensure homogenization of the newly added acid or base. Once shaken, the sample pH can be verified using narrow range pH paper.

10.9.3 Record any pH adjustments that have been made in the bench sheet and add the appropriate NCM when necessary.

10.10 Rinse the internal walls of the sample bottle with 20 mL of Methylene Chloride for quantitative transfer. Transfer this Methylene Chloride rinse to the separatory funnel for the initial extraction.

10.11 Seal the separatory funnels and rotate a few times. Vent all separatory funnels away from yourself to release the pressure. Continue rotating the separatory funnels for an additional 2 minutes.

10.12 Allow the organic solvent layer to separate from the water for a minimum of 10 minutes.

10.13 Drain the solvent layer through a powder funnel with sodium sulfate and collect the extract in a Turbo Vap vessel.

10.13.1 If an emulsion occurs so that it is 1/3 the solvent layer, employ manual techniques to complete the phase separation. Techniques that may be used are: centrifugation, pour backs or stirring the sample. Collect the emulsion layer in a 100mL glass centrifuge tube.

10.13.2 Following any manual technique used to break up the emulsion, place the aqueous layer back in the separatory funnel and pour the $MeCl_2$ layer into the powder funnel to collect the extract.

10.13.3 Rinse the centrifuge tube with 5-10 mLs of $MeCl_2$ and add this to the powder funnel to complete the transfer and collection.

10.13.4 Add the appropriate NCM in the LIMs system regarding the techniques employed to break up the emulsion.

10.14 Rinse each powder funnel with approximately 5-10 mL of methylene chloride after draining the solvent layer.

10.15 Perform two more extractions (repeating steps 10.10 through 10.14) with 20 mL of methylene chloride, shaking or rotating the separatory funnels for 1 minute each time. Rinse the powder funnel with 5-10 mL of methylene chloride after the third extraction.

10.16 Collect the solvent extracts in a Turbo Vap vessel or a French square.

10.17 Pour the extracted samples into the satellite “W” waste containers. Adjust the pH of each waste container to be between 5 and 9 and discard in the main “W” waste drum.

10.18 Concentrate the extracts using Turbo Vaps with Nitrogen blow down. Important: only concentrate approximately 150 mL of extract in the Turbo Vap vessel to avoid splashing of the sample and the risk of cross-contamination.

10.19 Keep the Turbo Vap nitrogen pressure as high as possible (20-25 psi) without splashing the extract.

- Water temperature should be maintained between 30-40°C.
- Splashing of the extracts must be avoided since cross-contamination could occur. Aluminum tins may be used to cover Turbo Vap vessels to assist in the prevention of cross contamination and analyte loss.
- During concentration, rinse the walls of the Turbo Vap vessels several times with a small amount of methylene chloride or appropriate solvent to push analytes back into the solvent and ensure quantitative collection.

10.20 Remove the Turbo Vap vessel from the Turbo Vap as soon as the 1.0 mL calibration mark is reached.

- Samples should be closely monitored to ensure that time spent in the Turbo Vap is minimized.
- Evaporation that exceeds 1.0mL results in the loss of analytes.

10.21 Solvent exchange samples to hexane if required by adding 10-20 mls of hexane and concentrating back to the 1.0mL mark. Refer to Table 3 for specific test requirements.

10.22 Homogenize the extract and perform any necessary cleanup procedures. Adjustment to the appropriate final volume may be done either before or after any required cleanup procedures, depending on the procedures to be performed. Reference the individual cleanup SOP for that information.

10.23 Bring samples to appropriate final volumes.

10.23.1 Final volume of 1.0mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel. Transfer the entire extract into a 2-mL vial using a 9 inch disposable pipette.

10.23.2 Final volume of 2.0mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel, add 1.0mL of final solvent using a repipetter. Transfer the entire extract to a labeled 4 mL vial using a 9 inch disposable pipette.

10.23.3 Final volume of 10.0 mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel, add 9.0mL of the final solvent to the vessel using a repipetter. Transfer 10.0mL into a labeled 40mL vial. Using a disposable pipette, transfer approximately 1.0mL to a labeled 2-mL vial. Retain extra volume for a period no less than 30 days.

10.23.4 Mark the meniscus on all extract vials for the analytical groups.

10.24 Method Specific Extractions

10.24.1 Pesticide (8081)

- Requires 3 extractions with the sample pH in the range of 5-9
- Type of water used for QC samples is Distilled water (DI)

- Extracted samples are solvent exchanged to Hexane
- Subject to Florisil Cleanup
- Final volume of extracts is 2.0 mLs

10.24.2 Polychlorinated Biphenyls (8082)

- Require 3 extractions with the sample pH in the range of 5-9
- Type of water used for QC samples is Distilled water (DI)
- Extracted samples are solvent exchanged to Hexane
- Subject to Silica Gel Cleanup (method 3630C) procedures
- Acid cleanup (method 3665A) is performed on all 8082 samples and associated QC
- Final volume for 8082 extracts is 2.0 mLs

10.24.3 Diesel Range Organics (310.13, 8015 DRO)

- Require 3 extractions with the sample pH <2.
- Type of water used for QC samples is Distilled water (DI).
- Final volume is 1.0 mL
- The oily nature of the spike and surrogate used for this extraction may result in a greater affinity for the Teflon sep funnels rather than the aqueous matrix.
- If low analyte recoveries are observed, 40 mLs of methylene chloride can be added to the empty separatory funnel for extraction. Rotate the separatory funnel for approximately thirty seconds and drain through the powder funnel. Concentrate this with the rest of the extract for that sample.

10.24.4 Semivolatile BNA (8270)

- Require 6 extractions
- 3 extractions are performed with the sample pH <2
- 3 extractions are performed with the sample pH >11
  Care should be taken to add only as much acid or base that is necessary to bring the sample within required range. Over acidifying samples results in the loss of Base/Neutral compounds. Over hydrolyzing samples results in the hydrolysis of compounds.
- Type of water used for QC samples is DI water
- Final volume is 1.0 mL

11 Calculations / Data Reduction N/A

12 Method Performance

Acceptable performance is monitored through the use of Method Detection Limit Studies as well as recoveries of surrogate and spike compounds.

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

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 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:

- The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- The analyst must read and understand this SOP.
- The analyst must read and understand the Method used as reference for this SOP.
- The analyst must complete a DOC or successfully analyze PT samples annually.

13 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).

14 Waste Management

14.1 The following waste streams are produced when this method is carried out.

14.1.1 Methylene Chloride waste. (Spent solvents are stored in satellite "C" waste containers. When full, a designated sample custodian will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.2 Hexane waste. (Spent solvents are stored in satellite "C" waste containers. When full, a designated sample custodian will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.3 Assorted flammable solvent waste from various rinses. (Spent solvents are stored in satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.4 Vials containing extracts in solvents. (Extract vials are disposed in BV waste drums and stored in the GC and GCMS SVOA departments. These drums are disposed of according to all state and federal regulations).

14.1.5 Unused sample volume should be discarded into "A" waste containers.

14.1.6 Extracted water samples. This material must be neutralized before it is discharged. (All extracted water shall be neutralized and dumped into the designated drum marked as "W" waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations).

14.1.7 Extracted aqueous samples contaminated with methylene chloride. This material must be neutralized before it is discharged to a POTW. (All extracted water shall be neutralized and dumped into the designated drum marked as "W" waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations).

14.1.8 Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. (Solid wastes are dried in trays inside a fume hood then transferred to 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste, sodium sulfate and glass wool or filter paper) are marked as "BC waste. When full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon

drum. This material will be disposed of according to all state and federal regulations.).

14.1.9 Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue. (All disposable glassware is dried of all solvents inside a fume hood then disposed of in a recycling bin).

14.2 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste management and Pollution Prevention."

15 References / Cross-References

15.1 Method 3510C, "Separatory Funnel Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Final Update III, December 1996.

15.2 MassDEP, Overview of the Analytical Data Enhancement Process for the Massachusetts Contingency Plan, WSC-CAM, Rev 1, July 2010

15.3 State of Connecticut, DEP, Laboratory Quality Assurance and Quality Control Guidance, RCP Guidance Documents, December 2010

16 Method Modifications:

16.1 The initial volume used for the 3510C method has been modified from 1000mL to 250mL. MDL and DOC studies have been performed and documented with the TA Buffalo QA department to verify that reporting limits for the analytical methods are still being achieved by utilizing this volume change.

17 Attachments

Table 1: Test Requirements Reference
Attachment 1: Example of current Bench Sheet

18 Revision History

Revision 0, dated 5 February 2013

- Integration for TestAmerica operations.

Table 1 Test Requirements Reference

Method	# Extractions	Initial pH	Secondary pH	QC Water Type	Hex. Exchange	Final Volume	Cleanup
8081	3	5-9	N/A	DI	Yes	2 mL	Florisil
8082	3	5-9	N/A	DI	Yes	2 mL	Silica Gel/Acid
8015B/310.13	3	<2	N/A	DI	No	1 mL	N/A
8270	6	<2	>11	DI	No	1 mL	N/A

Attachment 1: Example of Current Bench Sheet (Page 1 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402
Method Code: 480-3510C_LVI-480

Analyst: Dwyer, Nicole

Batch Open: 1/29/2013 2:24:07PM
Batch End:

Liquid-Liquid Extraction (Separatory Funnel)

	Input Sample Lab ID (Analytical Method)	SDG	GrossWt TareWt	InitAmnt FinAmnt	PHs Rcvd	PHs Adj1	PHs Adj2	Due Date	Analytical TAT	Div Rank	Comments	Output Sample Lab ID
1	MB-480-101402/1 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
2	MDLS-480-101402/2 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
3	MDLS-480-101402/3 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
4	MDLS-480-101402/4 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
5	MDLS-480-101402/5 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
6	MDLS-480-101402/6 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
7	MDLS-480-101402/7 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
8	MDLS-480-101402/8 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
9	MDLV-480-101402/9 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		
10	MDLV-480-101402/10 N/A	N/A	NA g NA g	250 mL 1 mL	7	<2	>11	N/A	N/A	N/A		

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TestAmerica Buffalo

Attachment 1: Example of Current Bench Sheet (Page 2 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402 Analyst: Dwyer, Nicole Batch Open: 1/29/2013 2:24:07PM

Method Code: 480-3510C_LVI-480 Batch End:

11	480-31887-A-1 (8270C)	N/A						1/28/13	10_Days - R	1	Not a sample. MDLS Tracking purposes only.

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Attachment 1: Example of Current Bench Sheet (Page 3 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402
Analyst: Dwyer, Nicole
Batch Open: 1/29/2013 2:24:07PM
Method Code: 480-3510C_LVI-480
Batch End:

Batch Notes

Person's name who did the prep	TG ND
Prep Solvent Name	MeCl2
Prep Solvent Lot #	0000027258
Prep Solvent Volume Used	120
Person's name who witnessed reagent drop	TG
Acid used for pH adjustment	1:1 Sulfuric Acid
Acid used for pH adjust Lot #	2032711
Base used for pH adjustment	Sodium Hydroxide
Base used for pH adjust Lot #	HC257086
Silica Gel Lot Number	NA
Person's name who did the concentration	ND/TG
Exchange Solvent Name	NA
Exchange Solvent Lot #	NA
Concentration Start Time	NA
Concentration End Time	NA
Final Concentrator Volume	1
Na2SO4 Lot Number	27863001
ID number of the thermometer	NA
Uncorrected Temperature	NA
Oven, Bath or Block Temperature 1	NA
N-evap #	NA

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Attachment 1: Example of Current Bench Sheet (Page 4 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402 Analyst: Dwyer, Nicole Batch Open: 1/29/2013 2:24:07PM

Method Code: 480-3510C_LVI-480 Batch End:

N-evap temperature	NA
Uncorrected N-evap Temperature	NA
Sufficient volume for MS/MSD?	NO
Florisil Lot #	NA
TBA Lot #	NA
Copper Lot #	NA
Acid used for Clean Up Reagent	NA
Pipette ID	NA
Syringe Lot #	NA
pH Paper Lot Number	NA
Filter Paper Lot Number	NA
Glass Wool ID	NA
NaCl Lot #	NA
Batch Comment	Large Volume Injection for 8270/ 3510C

Comments

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Attachment 1: Example of Current Bench Sheet (Page 5 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402 Analyst: Dwyer, Nicole Batch Open: 1/29/2013 2:24:07PM

Method Code: 480-3510C_LVI-480 Batch End:

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
MDLS 480-101402/2	MB_ASLVIM_WRK_00001	1 mL	1 mL	ND	TL
MDLS 480-101402/2	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/2	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLS 480-101402/3	MB_ASLVIM_WRK_00001	1 mL	1 mL		
MDLS 480-101402/3	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/3	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLS 480-101402/4	MB_ASLVIM_WRK_00001	1 mL	1 mL		
MDLS 480-101402/4	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/4	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLS 480-101402/5	MB_ASLVIM_WRK_00001	1 mL	1 mL		
MDLS 480-101402/5	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/5	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLS 480-101402/6	MB_ASLVIM_WRK_00001	1 mL	1 mL		
MDLS 480-101402/6	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/6	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLS 480-101402/7	MB_ASLVIM_WRK_00001	1 mL	1 mL		
MDLS 480-101402/7	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/7	MB_LVIMDL_WRK_00001	1 mL	1 mL		

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Attachment 1: Example of Current Bench Sheet (Page 6 of 6)

Aqueous Extraction Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-101402 Analyst: Dwyer, Nicole Batch Open: 1/29/2013 2:24:07PM

Method Code: 480-3510C_LVI-480 Batch End:

MDLS 480-101402/8	MB_A9LVIM_WRK_00001	1 mL	1 mL	NO	TG
MDLS 480-101402/8	MB_ALVIMD_WRK_00001	1 mL	1 mL		
MDLS 480-101402/8	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLV 480-101402/9	MB_LVIMDL_WRK_00001	1 mL	1 mL		
MDLV 480-101402/10	MB_A9LVIM_WRK_00001	0.5 mL	1 mL		
MDLV 480-101402/10	MB_ALVIMD_WRK_00001	0.5 mL	1 mL		
MDLV 480-101402/10	MB_LVIMDL_WRK_00001	0.5 mL	1 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

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Ultrasonic Extraction of Soils and Wipes (Method No 3550B)

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

Michelle Freeman	7/10/2013 Date	Kenneth Kasperek	7/10/2013 Date
Department Manager		EHS Manager	
Brad Prinzi	7/10/2013 Date	Christopher Spencer	7/10/2013 Date
Quality Assurance Manager		Laboratory Director	

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes. The ultrasonic process used ensures thorough contact of the sample with the extraction solvent.

2.0 Analytes, Matrix(s), and Reporting Limits

2.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes.

2.2 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.

3.0 Summary of Method

3.1 Low Level

A 30 gram sample is mixed with anhydrous sodium sulfate. This is solvent extracted three times using ultrasonic extraction. The extract is then filtered and concentrated. The extract may then be subject to clean-up procedures or sent directly for analysis.

3.2 Medium/High Level

A 2 gram sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis. (For Caulks – use 0.10 gram sample size and solvent extract once using ultrasonic extraction).

3.3 Wipes

A wipe sample is mixed with anhydrous sodium sulfate and solvent extracted using ultrasonic extraction.
For 8082 PCB, a portion of the extract is removed for cleanup and/or analysis.

4.0 Definitions

Standard definitions are found in Section 3.2 of the Laboratory Quality Manual

5.0 Interferences

5.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks. Matrix interference may be caused by contaminants that are co-extracted from the sample.

5.2 Major organic interferences may be removed during cleanup procedures

6.0 Safety

6.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

6.2 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.

6.3 Specific Safety Concerns or Requirements

6.3.1 All parameters of this extraction where solvent is being used must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system.

6.3.2 Any excess non extracted sample (including dry weights) waste will be disposed of in “BE” waste. Solid waste generated in the extraction process will be disposed of in “BC” waste. All solvent and extract waste is disposed of in “C” waste.

6.3.3 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used for extractions and should not be used.

6.3.4 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

6.3.5 Due to the high frequency produced from the sonicators, it is necessary to utilize both hood sashes to keep the noise level to a minimum.

6.4 Primary Materials Used

6.4.1 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 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	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.
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.
		1 – Always add acid to water to prevent violent reactions.	
		2 – Exposure limit refers to the OSHA regulatory exposure limit.	

7.0 Equipment and Supplies

7.1 Aluminum Dishes, Foil
7.2 Disposable, wood tongue depressor
7.3 Toploader Balance, capable of accurately measuring to 2.0g
7.4 Syringes
7.5 ¾ in. dual horn Sonicators® with Sonabox® acoustic enclosures
7.6 16 oz. french squares, disposable
7.7 Ovens - 104°C and 400°C
7.8 16 oz. wide mouth jars, disposable
7.9 Turbovap concentrators and vessels
7.10 Stainless steel filter funnels
7.11 Graduated cylinders
7.12 Ear Protection
7.13 2,10 and 25 or 40 ml vials, septa and caps
7.14 Disposable pipettes and pipette bulbs
7.15 18.5 cm #41 filter paper
7.16 Microtip horn Sonicators with Sonabox acoustic enclosures.

8.0 Reagents and Standards

8.1 All solvents are pesticide grade or equivalent.
8.2 Hexane delivered in cycletainers
8.3 Compressed Nitrogen

8.4 Anhydrous granular sodium sulfate, previously baked in a 400°C oven for a minimum of 4 hours, cooled and dried in a dessicator, and rinsed with methylene chloride. Or purchased pre-baked from Jost chemical.
8.5 Methylene Chloride delivered in cycletainers
8.6 Acetone delivered in cycletainers
8.7 Surrogate and spike solutions appropriate to the analytical method.
8.8 De-ionized water (DI)

9.0 Sample Collection, Preservation, Shipment and Storage

9.1 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.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	30 grams	Cool 4 ± 2°C	14 Days from sample	SW-846, third edition
Soils	Glass	30 grams	Cool 4 ± 2°C	10 days from receipt	CLP

10.0 Quality Control

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1in 20 or fewer samples	< Rpt. Limit
Laboratory Control Standard (LCS)[1]	1in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1in 20 or fewer samples	Statistical Limits [4]
Matrix Spike Duplicate (MSD)[2]	1in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

10.1.1 Laboratory Control Standard Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
10.1.2 The sample selection for MS/MSD is randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
10.1.3 Analytical and QC samples (MB, LCS,LCSD, MS/MSD)
10.1.4 Statistical control limits are updated annually and are updated into LIMS.

11.0 Procedure

Sample volumes and Bottle IDs are directly recorded into LIMs bench sheets.

11.1 Low Level Extraction:

11.1.1 Decant and discard any standing water on the sample. Enter the appropriate NCM into LIMS if sample was decanted. Discard any sticks, leaves, rocks or other foreign matter.

11.1.2 Tare a labeled 16oz wide mouth jar and transfer 30 grams of homogenized soil. For blank samples (LCS,LCSD, or MB), approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire extraction procedure

11.1.3 Add granular sodium sulfate to the 30g sample and blend with a wood tongue depressor until the sample is free flowing.

11.1.4 Add surrogate to the samples using the appropriate surrogate as designated per the analytical

11.1.5 Add the appropriate spike to the QC samples (LCS/LCSD/MS/MSD) as designated per the analytical method.

11.1.6 Immediately add 100mls of appropriate solvent to the sample; the solvent for determinative methods is as follows:

All CLP methods –
1:1 methylene chloride/acetone: Combine 500 ml of methylene chloride and 500 ml of acetone in a clean 1 liter glass amber bottle.

8081,8082 - 1:1 acetone/hexane: Combine 500 ml of acetone and 500 ml of hexane in a clean 1liter glass amber bottle.

8270 – 1:1 Methylene Chloride:Acetone

8015DRO - Methylene chloride

*8270 soils for specific clients (NiSource) will be extracted with 1:1 Methylene chloride/Acetone

11.1.7 Fold an 18.5cm filter paper into quarters and place it in a stainless steel filter funnel. Place this funnel in a labeled french square bottle.

11.1.8 Before use, clean the sonication horns with 12.5% Nitric acid rinse, DI water, acetone, and the solvent the extraction calls for. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.1.9 Place the 16oz. wide mouth jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch

and still above the soil sample by the ½ inch. In the case of excessively wet samples that needed a great deal of sodium sulfate, more solvent may be added and the position of the sonicator jar adjusted to the ideal parameters.

11.1.10 Sonicate for 3 minutes at out put setting 7-10, pulsed mode, 50% duty cycle, using ¾ inch horn.

11.1.11 Collect the extract in a labeled french square jar by first decanting the extract through the filter funnel containing the 18.5 cm filter paper folded inside. Rinse the filter paper with the appropriate extraction solvent after the first sonication round is decanted. When using solvents with acetone, add a little sodium sulfate to the filter paper to reduce the amount of water in the extract.

11.1.12 Repeat steps 11.1.9-11.1.11 twice more.

11.1.13 After the third sonication, rinse the contents of the sonication jar into the funnel.

11.1.14 After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used. Allow the sample to drain completely inside a fume hood.

11.1.15 Clean the sonicator horn between samples as describe in section 11.1.8.

11.2 Concentration Procedure

11.2.1 Pour the extract into a labeled turbovap vessel that has been pre-rinsed with $MeCl_2$, rinse the french square with the appropriate solvent and add this to the turbovap vessel.

11.2.2 Place the vessel in the turbovap and turn on the nitrogen to concentrate the extract to 1ml. During concentration, the turbovap vessel should be periodically rinsed with the extraction solvent. The temperature of the turbovap water bath must be maintained between 30˚C and 40˚C.

11.2.3 For 8270 and 8015 DROs, concentrate to a final volume of 1ml using the calibrated 1.0ml mark on the turbovap vessel. Transfer entire volume to a 2ml vial using a disposable 9-inch pipette and mark the meniscus. 8270 samples can be relinquished to GC/MS for analysis and DRO samples can be relinquished to GC for analysis.

11.2.4 For 8081/8082 concentrate the extract to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel. Adjust the final volume to 10.0ml by adding 9.0ml of Hexane to the turbovap vessel with a repipettor. If cleanup of samples is required, refer to specific cleanup SOP. If no cleanup is required, transfer 1 ml using a disposable pipette to a 2ml vial. Mark the meniscus on the vial and relinquish to GC for analysis. Transfer remaining extract volume to an appropriately labeled 40ml vial and store for no less than 30 days. All 8082 extracts go through sulfuric acid cleanup. See Sulfuric Acid Cleanup SOP for details.

11.2.5 For all CLP method soils, GPC cleanup is required. Bring the volume to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel then adjust the final volume to 10.0ml by adding 9.0ml of Methylene chloride to the turbovap vessel with a repipettor. Transfer to a 40mL vial. Cap and set aside in a $4^{o}C \pm 2^{o}$ C incubator for later clean up by GPC. See GPC SOP for GPC procedure.

11.3 MEDIUM LEVEL EXTRACTION:

No dry weight is required for caulk samples, and samples containing asbestos. The dry weight will be recorded as 100% dry in LIMS.

The sample volume used for caulk samples is 0.10-0.19 grams. This reduced volume aids in minimizing contamination commonly seen from caulk samples.

11.3.1 Decant and discard any standing water on the sample. Enter the appropriate NCM into LIMS if sample was decanted. Discard any sticks, leaves, rocks or other foreign matter.

11.3.2 Transfer 2 grams of the homogenized sample into a tarred 25 mL extraction vial. For QC samples (LCS, LCSD, or MB), approximately 2 g of sodium sulfate will be used in lieu of soil and shall be taken through the entire extraction procedure.

11.3.3 Add granular sodium sulfate to the 2g sample and blend with a disposable tongue depressor until the sample is free flowing.

11.3.4 Add surrogate to the samples using the appropriate surrogate per the analytical method. Write an “X” on the label after adding the surrogate.

11.3.5 Add the appropriate spike to the QC samples (LCS/LCSD/MS/MSD) Circle the “X” after adding the appropriate spike.

11.3.6 Bring the volume of the sample to 10mL using 8 or 9 mLs of hexane (depending on the sample).

11.3.7 Before use, clean the sonication horns with 12.5% Nitric acid rinse, DI water, acetone, and hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.3.8 Sonicate each sample once for 45 seconds on pulse mode and setting of 5 using a microtip sonicating horn.

11.3.9 Decant the sample into a 40mL vial that is pre labeled with the appropriate sample ID number. Aliquot 1.0ml using a disposable pipette in a 2mL vial, mark the meniscus and relinquish to GC for analysis. The remaining extract is to be saved for no less than 30 days. 8082 extraction requires acid cleanup. Refer to SOP BF-OP-010.

11.3.10 Clean the sonicator horn between samples as describe in section 13.3.7.

11.4 Wipe Extraction

11.4.1 Place entire sample into a labeled 8oz. wide-mouth jar.

11.4.2 Add anhydrous granular sodium sulfate.

11.4.3 Add 1ml of specific wipe surrogate.

11.4.4 Add appropriate spike to QC samples (LCS/LCSD/MS/MSD)

11.4.5 For QC samples (LCS, LCSD, and MB), approximately 30g of sodium sulfate will be used in lieu of wipe and shall be taken through the entire analytical procedure.

11.4.6 **For 8082 Wipes:** Add hexane so that the total final volume is 40 mls taking into account the volume of spike and surrogate added.

11.4.7 Clean the sonicator horns before beginning extraction, and between samples with 12.5% Nitric Acid rinse, DI water, acetone, hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.4.8 Place the 8oz. wide mouth jar under the sonicator horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch.

11.4.9 Sonicate for 1.5 minutes at out put setting 8 or 9, pulsed mode, 50% duty cycle, using ¾ inch horn.

11.4.10 Transfer approximately 10 mls of solvent to an appropriately labeled 40 ml vial. 8082 extraction requires acid cleanup. Refer to SOP BF-OP-010 Transfer 1.0 ml using a disposable pipette to 2ml vial, mark the meniscus, and relinquish to the appropriate analytical lab. The remaining extract is to be saved for no less than 30 days. Record the final volume as 40.0mls in LIMs.

11.4.11 **For 8015DRO Wipes**:

11.4.12 After adding 1.0mL of spike and surrogate, add 100mLs of Methylene Chloride to the sample jar

11.4.13 Place the 8oz. wide mouth jar under the sonicator horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch.

11.4.14 Sonicate for 3 minutes at out put setting 7-10, pulsed mode, 50% duty cycle, using ¾ inch horn.

11.4.15 Collect the extract in a labeled french square jar by first decanting the extract through the filter funnel containing the 18.5 cm filter paper folded inside.

Rinse the filter paper with the appropriate extraction solvent after the first sonication round is decanted. When using solvents with acetone, add a little sodium sulfate to the filter paper to reduce the amount of water in the extract.

11.4.16 Repeat steps 11.4.12-11.4.15 twice more.

11.4.17 After the third sonication, rinse the contents of the sonication jar into the funnel.

11.4.18 After the sample has drained, rinse down the funnel with additional extraction solvent. Allow the sample to drain completely inside a fume hood.

11.4.19 Clean the sonicator horn between samples as describe in section 11.4.7.

11.4.20 Pour the extract into a clean, labeled turbovap vessel. Rinse the french square with the appropriate solvent and add this to the turbovap vessel.

11.4.21 Place the vessel in the turbovap and turn on the nitrogen to concentrate the extract to 1ml. During concentration, the turbovap vessel should be periodically rinsed with the extraction solvent. The temperature of the turbovap water bath must be maintained between 30˚C and 40˚C.

11.4.22 For 8015 DROs, concentrate to a final volume of 1.0mL using the calibrated 1.0mL mark on the turbovap vessel. Transfer entire volume to a 2.0mL vial using a disposable 9-inch pipette and mark the meniscus.

11.5 Calibration

11.5.1 Analytical Balances are checked on a daily basis, and calibrated by a NIST certified company.

11.5.2 Sonicator horns are checked on a daily basis and tuned at least every 6 months.

12.0 Calculations / Data Reduction N/A

13.0 Method Performance

13.1 Acceptable performance is monitored through the use of Method Detection Limit Studies, as well as, recoveries of surrogate and spike compounds.

13.2 Method Detection Limit Study (MDL)

13.2.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in 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.

13.3 Demonstration of Capabilities

Refer to Buffalo Quality Laboratory Manual

13.4 Training Requirements

Refer to Buffalo Quality Laboratory Manual.

14.0 Pollution Control

14.1 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).

15.0 Waste Management

15.1 The following waste streams are produced when this method is carries out.

15.1.1 Waste hexane. Extra volume is stored for a period of no less than 30 days. After 30 days the waste is disposed of according to all state and federal regulations.

15.1.2 Waste Methylene Chloride, Acetone, and/or Hexane. Spent solvents are stored in satellite "C" waste containers. When full satellite containers will be transferred to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations.

15.1.3 Waste solid material from the extraction process. Solid Wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When full the satellite containers will be transferred into a 55-gallon drum and disposed of according to all state and federal regulations.

15.1.4 Used sodium sulfate, glass wool, or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. Lab generated solid wastes (extracted solid waste) are marked as "BC waste". When full the satellite containers will be transferred into a 55-gallon drum and disposed of according to all state and federal regulations.

15.1.5 Assorted flammable solvent waste from various glassware rinses. Spent solvents are stored in satellite "C" waste containers. When full satellite containers will be transferred to a grounded 55-gallon drum. These are

located in the secured waste area and are disposed of according to all state and federal regulations.

15.1.6 Miscellaneous disposable glassware contaminated with solvents and sample residue. All disposable glassware contaminated with solvent is air dried inside an operational fume hood then disposed in the recycling receptacle.

15.1.7 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."

16.0 References / Cross-References

16.1 USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, Third Edition; Revision 2, December 1996; Method 3550B.

16.2 USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLMO4.3.

17.0 Method Modifications:

Item	Method xx	Modification
1	3550B 7.2	Dry weights for samples are kept in a 104 C for 3 hours..
2	3550B 7.3.4	Samples are allowed to gravity drain through filter paper to minimize analyte recovery loss.
3	3550B 7.4.1	Sample volume used for medium level Caulk extraction is reduced from 2 grams to 0.1-0.2 grams to minimize contamination commonly seen from this matrix.

18.0 Attachments

18.1 Attachment 1: Organic Prep Worksheet

19.0 Revision History

- Revision 4, dated 10, July 2013
  - Updated Section 11.4 to include procedure for 8015DRO wipe extraction
  - Changed Quality Manager, signature added

- Revision 3, dated 21, December 2012
  - Updated Section 11.1.6 for 8270 solvent system

- Revision 2, dated 13, January 2012
  - Update LIMs verbiage
  - Updated prep directions for caulk extraction in Section 11.3.
  - Removed outdated spike/surrogate recipe sheets.
  - Updated Worksheet Attachment
  - Changed Department and Quality Managers, signature added.

- Revision 1, dated 12, March 2010
  - Updated nomenclature of QC samples
  - Included process for Sulfuric acid cleanup for all 8082 extractions for Section 11.2.4 (removed interim form)
  - Removed dry-weight references in Section 11.0.
  - Removed sections 11.2.6, 11.2.7 concerning GPC cleanup
  - Updated vial sizes
  - Updated Table 1
  - Updated spike/surrogate amount for wipe procedure in Section 11.0.
  - Added Nitric Rinse for cleaning horns between extractions in Section 11.0.
  - Updated power mode and duration settings for sonicators
  - Changed sample volume used for Caulk samples from 2 grams to 0.1-0.2 grams.
  - Updated Method Modifications table to include reduced volume for Caulk samples.
  - Included comment in section 11.0 clarifying bench sheets used for soil extractions.

- Revision 0, dated 24, January 2008
  - Integration for TestAmerica and STL operations.
  - Updated section 13.1.8 to include recipe for making 1:1 methylene chloride/acetone and 1:1 acetone:hexane
  - Updated Table 1,2, and 3

Attachment 1: Organic Prep Worksheet

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-47529 Analyst: Myers, Carmela Batch Open: 1/11/2012 8:10:27AM

Method Code: 480-3550B-480 Batch End:

Ultrasonic Extraction

	Input Sample Lab ID (Analytical Method)	SDG	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments	Output Sample Lab ID
1	MB-480-47529/1 N/A	N/A	+30.29 g	10 mL	N/A	N/A	N/A		
2	LCS-480-47529/2 N/A	N/A	+30.27 g	10 mL	N/A	N/A	N/A		
3	LCSD-480-47529/3 N/A	N/A	+30.60 g	10 mL	N/A	N/A	N/A		
4	480-14789-B-1 (8081A)	N/A	+30.30 g	10 mL	1/12/12	3_Day_RUSH -	4		
5	480-14791-B-1 (8081A)	N/A	+30.22 g	10 mL	1/12/12	3_Day_RUSH -	4		
6	480-14791-B-2 (8081A)	N/A	+30.46 g	10 mL	1/12/12	3_Day_RUSH -	4		

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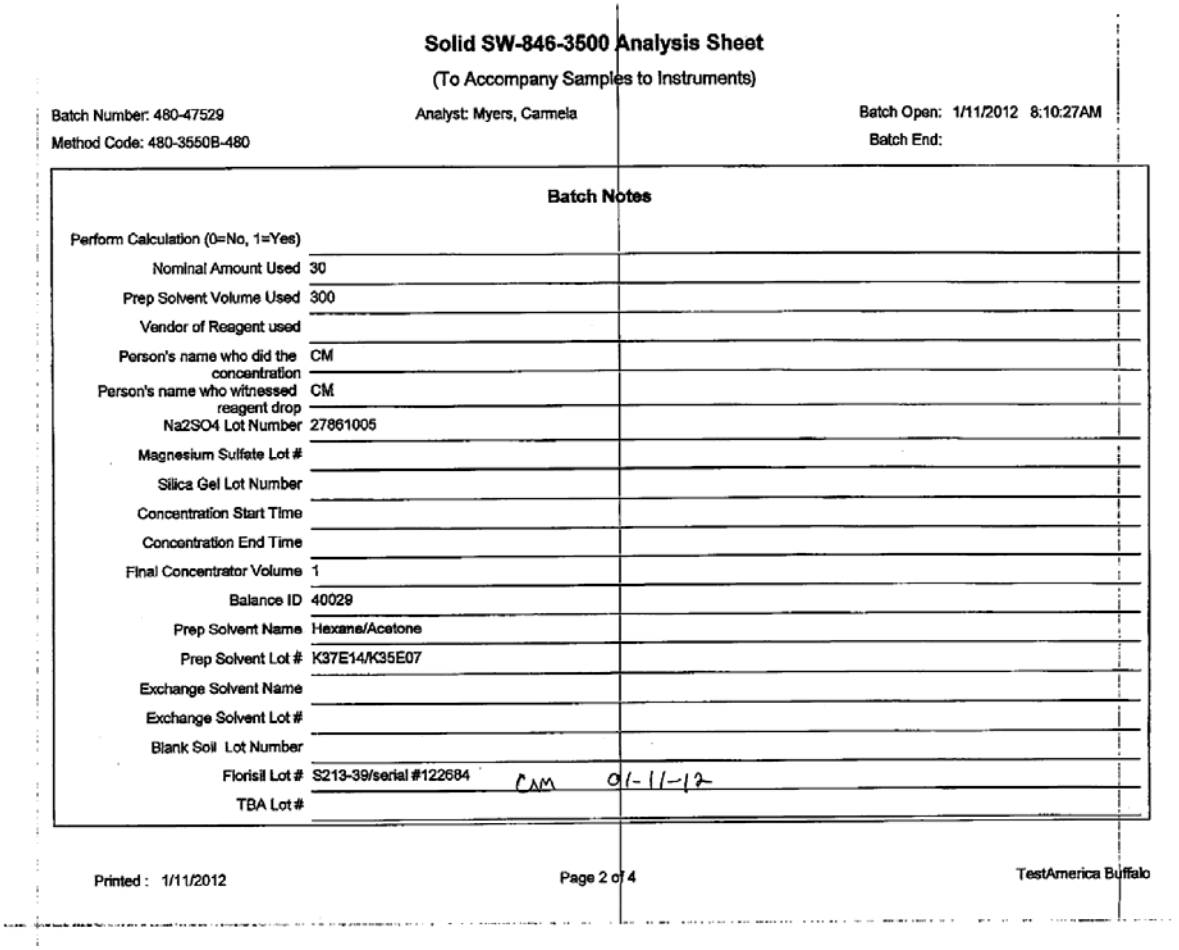

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-47529
Method Code: 480-3550B-480

Analyst: Myers, Carmela

Batch Open: 1/11/2012 8:10:27AM
Batch End:

Batch Notes

Perform Calculation (0=No, 1=Yes)	
Nominal Amount Used	30
Prep Solvent Volume Used	300
Vendor of Reagent used	
Person's name who did the concentration	CM
Person's name who witnessed reagent drop	CM
Na2SO4 Lot Number	27861005
Magnesium Sulfate Lot #	
Silica Gel Lot Number	
Concentration Start Time	
Concentration End Time	
Final Concentrator Volume	1
Balance ID	40029
Prep Solvent Name	Hexane/Acetone
Prep Solvent Lot #	K37E14/K35E07
Exchange Solvent Name	
Exchange Solvent Lot #	
Blank Soil Lot Number	
Florisil Lot #	S213-39/serial #122684 CAM 01-11-12
TBA Lot #	

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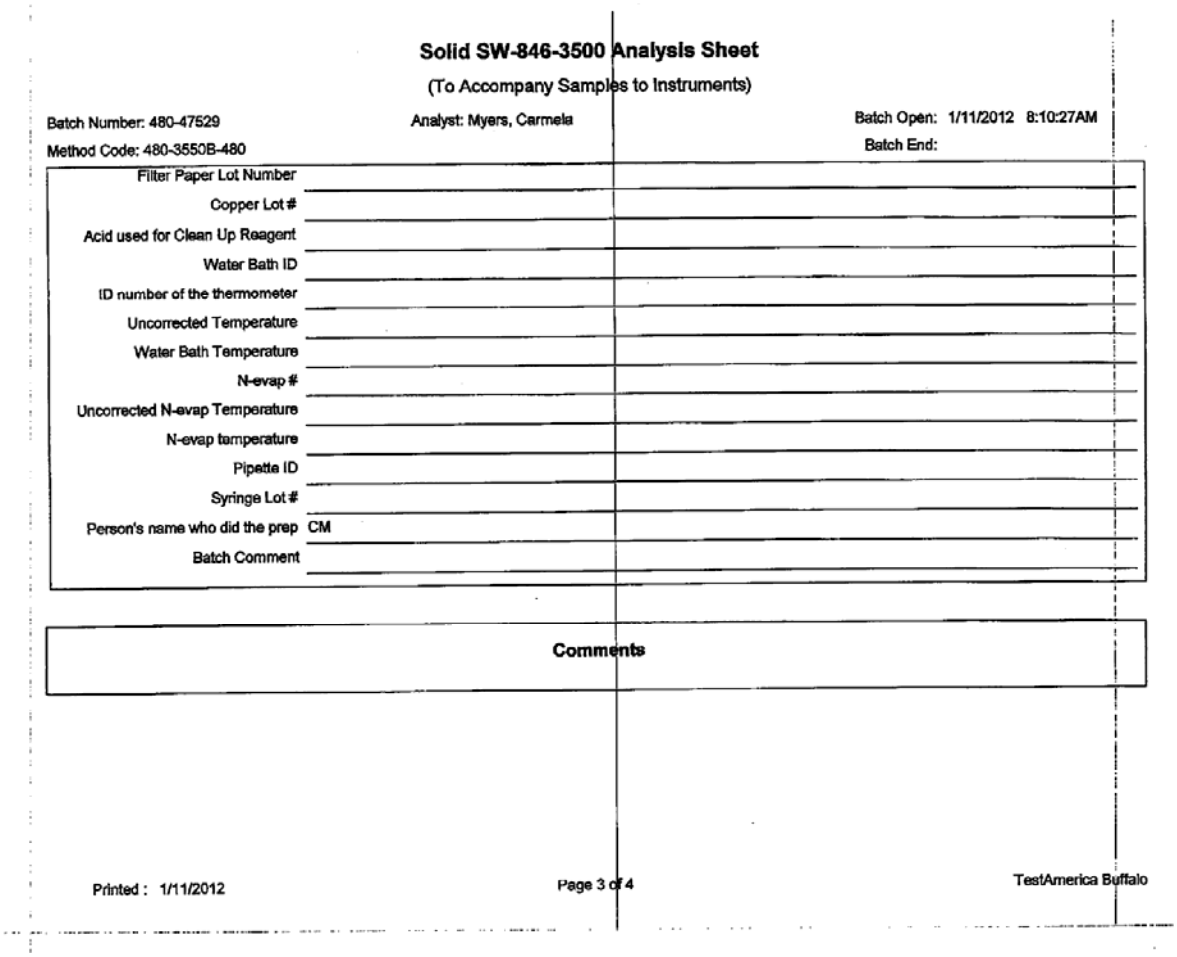

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-47529 Analyst: Myers, Carmela Batch Open: 1/11/2012 8:10:27AM

Method Code: 480-3550B-480 Batch End:

Filter Paper Lot Number	
Copper Lot #	
Acid used for Clean Up Reagent	
Water Bath ID	
ID number of the thermometer	
Uncorrected Temperature	
Water Bath Temperature	
N-evap #	
Uncorrected N-evap Temperature	
N-evap temperature	
Pipette ID	
Syringe Lot #	
Person's name who did the prep	CM
Batch Comment	

Comments

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Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-47529
Method Code: 480-3550B-480

Analyst: Myers, Carmela

Batch Open: 1/11/2012 8:10:27AM
Batch End:

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
MB 480-47529/1	O_8081/82surr_00016	1 mL	10 mL	CM	
LCS 480-47529/2	O_8081/82surr_00016	1 mL	10 mL		
LCS 480-47529/2	O8081pestspik_00008	1 mL	10 mL		
LCSD 480-47529/3	O_8081/82surr_00016	1 mL	10 mL		
LCSD 480-47529/3	O8081pestspik_00008	1 mL	10 mL		
480-14789-B-1	O_8081/82surr_00016	1 mL	10 mL		
480-14791-B-1	O_8081/82surr_00016	1 mL	10 mL		
480-14791-B-2	O_8081/82surr_00016	1 mL	10 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

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TestAmerica Buffalo

Title: Method 5030C: Purge and Trap

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Approvals (Signature/Date):

Denise Giglia — 7/30/13	Kenneth Kasperek — 7/30/13
Denise Giglia — Date	Kenneth Kasperek — Date
Department Manager	EHS Manager
Brad Prinzi — 7/30/13	Christopher A. Spencer — 7/30/13
Brad Prinzi — Date	Christopher A. Spencer — Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure. The gas chromatographic determinative steps are found in Methods 8260C, 624, 524.2, NYSDEC Analytical Services Protocols, and USEPA OLMO4.3.

1.1 Analytes, Matrix(s), and Reporting Limits

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

An inert gas, helium, is bubbled through a sample (solution) at ambient temperature or an elevated temperature depending on analytes and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After sample purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

3.0 Definitions

Standard definitions are found in TestAmerica Buffalo's Laboratory Quality Manual.

4.0 Interferences

Purchasing high-quality helium minimizes impurities from the purge gas (helium). The purge and trap system is highly susceptible to carryover from high level samples. Sample lines are flushed with volatile free water after each sampling. The trap is baked at 260 degrees C for a minimum of eight minutes.

The laboratory analyzes weekly volatile holding blanks to ensure an environment free of volatile organic solvent vapors. Methylene chloride can permeate through a septum seal, a trip blank is carried through the sampling and handling protocols to serve as a check on such contamination.

The purge and trap system will also be demonstrated to be clean by the use of Method Blanks and IBLKs. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Unusually high-concentration samples should be followed by an analysis of organic-free reagent water to check for cross-contamination.

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.

Specific Safety Concerns or Requirements

Special precautions are taken when working with a purge and trap system. Due to the amount of gas utilized by the system, all employees are required to wear approved safety glasses. Parts of the system are under pressure, always allowing for the possibility of shattered glass.

Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

Purge and trap device that consists of three parts.

Sample purge vessels are designed to accept 5ml samples and have a total volume of less than 15 mls. In low level drinking water methods, 25ml sample purge vessels are utilized.

A VOCARB 3000 trap (or similar manufacturers trap) ~30cm long containing the following materials is utilized for all methods:

10cm Carbopack B
6cm Carboxen 1000
1cm Carboxen 1001

The desorber rapidly pre-heats the trap to 245 degrees C and then desorbs at 250 degrees C. The trap is then baked at 260 degrees C.

7.0 Reagents and Standards

Volatile free water for making sample dilutions and method blanks.
Purge and trap grade methanol for standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Samples should be collected in 40 ml capped vials with zero headspace and stored at 4°C +/-2° until time of analysis. Aqueous samples preserved with HCl must be analyzed within 14 days of collection. Aqueous samples not preserved with HCl must be analyzed within 7 days of collection. Soil samples must be analyzed within 14 days of collection. TCLP volatile samples must be tumbled with in 14 days of collection and then analyzed within 14 days of the TCLP extraction.

9.0 Quality Control

A standard, LCS, and MB is analyzed in each run as well as a MS/SD every 20 samples.

9.1 Instrument QC

Instrument Operating Conditions (Suggested)

Purge temperature	<35-40ºC
Desorb Temperature	250ºC
Line Temperature	110ºC
Purge Gas (Helium)	40mL/min.
Purge Total Time	11 min.
Desorb Time	2 min.

Instrument Maintenance

Upon verification of established operating conditions, the following is performed on a sequence basis:

- check purge flow;
- analyze blank to insure system is free of contamination (daily);
- vessel and lines are flushed with volatile free water after each analysis.

Note: System must be leak free. System can be checked by purging 5mL water in sample vessel and capping off vent on purge device. If purge flow stops system is leak free, if purge flow continues (within 2-3 minutes) this means there is a leak within the system. Leak must be located and corrected.

10.0 Procedure

Instrument Operating Conditions (Suggested)

Purge temperature	<35-40ºC
Desorb Temperature	250ºC
Line Temperature	110ºC
Purge Gas (Helium)	40ml/min.
Purge Total Time	11min.
Desorb Time	2min.

10.1 Calibration

See appropriate determinative method(s).

11.0 Calculations

NA

12.0 Method Performance

MDLs are performed yearly, per analytical method, and kept on file with the Quality Department. 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 20.7 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.

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 BF-WM-01. The following waste streams are produced when this method is carried out:

14.1 Acidic material from the auto-sampler: Waste stream must be collected in "A" waste receptacles and neutralized before discharge to a sewer system.

14.2 Methanol waste from rinses and standards: Collect in "C" waste receptacles. In the case of medium level soil extractions, the methanol is decanted off the soil and collected in the "C" receptacle. Waste receptacles are then taken to sample control where they are disposed of properly. Excess samples (acidic and non-acidic). Collect in "A" waste receptacles and are neutralize before disposal into drain/sewer.

14.3 Excess soil sample from medium level extraction: Place in solid waste receptacle. Soils for dry weight measurements are also disposed in this manner.

15.0 References

U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984

U.S. EPA "Method 5030C, Purge and Trap for Aqueous Samples", Test Methods for Evaluating Solid Waste, Volume 1B, Revision 3, May 2003.

16.0 Tables, Diagrams, Flowcharts NA

17.0 Revision History

- Revision 0, July 30, 2013 initial release

Title: Method 5035A-L and 5035A-H – Closed System Purge & Trap and Extraction for Volatile Organics in Soil and Waste Samples

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

Denise Giglia 6/7/12 Denise Giglia Date Department Manager	Kenneth Kasperek 6/7/12 Kenneth Kasperek Date Technical Director
Paula M. Benham 6/7/12 Paula Benham Date Quality Assurance Manager	6/7/12 Christopher Spencer Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1. **Matrix**

1.1.1. Applicable matrixes are solid materials such as soils, sediments, and solid wastes.

1.2. **Reporting Limits**

1.2.1. GCMS Low Level Reporting limit = 5.0 ug/kg for most compounds
1.2.2. GCMS Medium Level Reporting Limit = 100 ug/kg
1.2.3. GC Low Level Reporting Limit = 1 ug/kg for most compounds
1.2.4. GC Medium Level Reporting Limit = 10 ug/kg for most compounds

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 This method describes a closed-system purge and trap process for the analysis of volatile organic compounds (VOC's) in solid materials (e.g. soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for high concentration VOCs and oily wastes. These procedures will be used in conjunction with the appropriate determinative gas chromatographic procedure (methods 8260, 8021, OLM04.3, ASP00 and ASP05).

2.2 The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the Atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are minimized.

2.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels > 200ug/kg.

2.4 Procedures are also given for addressing oily wastes that are soluble in a water miscible solvent. These samples are also purged using Method 5030.

3.0 Definitions

3.1 VOC - Volatile organic compound.

3.2 MB – Method Blank

3.3 IBLK – Instrument Blank

3.4 Standard definitions may be found in the TestAmerica Buffalo Laboratory Quality Manual.

4.0 Interferences

4.1 Volatile organic contaminates can occur from a number of sources. All solvent handling is accomplished under a hood to minimize organic vapors in the analytical laboratory. MBs and IBLKs will be utilized to demonstrate a clean system.

4.2 Since some organic volatile compounds can permeate through a sample septum seal, a Volatile Holding Blank will be stored with all samples and analyzed weekly by Method 8260.

4.3 Contamination by carryover can occur whenever a sample with high levels is analyzed. To reduce carryover, the purging device, syringe and lines are flushed between every analysis. The trap is baked at 260-270°C between each analysis.

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** None

5.2 **Primary Materials Used**

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and Trap devices -Autosampler, - Archon 5100a
6.1.1.1 Concentrator, - Encon or Eclipse Model 4660

6.1.2 Gas Chromatograph/Mass Spectrometer (GCMS)
6.1.2.1 GC, - Agilent HP6890
6.1.2.2 MS, - Agilent HP5973

6.1.3 Data System - Chemstation with Chrom software

6.2 Supplies

6.2.1 Syringes - Hamilton syringe size - 10ul, 25ul, 50ul, 250ul, 1ml, 5ml, 10ml & 25ml

6.2.2 Pasteur pipettes - disposable

6.2.3 Vials and Caps -40 ml disposable, 2ml disposable

6.2.4 Volumetric flasks - Pyrex 50ml, Pyrex 100ml

6.2.5 pH paper - wide range, Baxter Diagnostics Inc.

6.2.6 Analytical Balance - Toledo Inc. Mettler AE160

6.2.7 Traps - Supelco Vocarb 3000 or OI Trap #10

6.2.8 Gas Chromatograph Column - J&W Scientific DB-624

6.2.9 Magnetic stir bars

6.2.10 En Core extrusion tool

6.2.11 En Core samplers

6.2.12 Small freezer

7.0 Reagents and Standards

7.1 Reagent Water - Reagent water is prepared by passing water through a carbon trap.

7.2 Methanol - purge and trap grade

7.3 See appropriate determinative method for standards

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Arrival of at least three soil samples in EnCore ™ sampling devices, require appropriate storage within 48hrs. The soil from two of the EnCore™ devices are removed and immediately placed into tared 40ml vials. The sample weight is then recorded in the weight logbook for GC or directly uploaded from the scale into the preparation batch in TALS for GCMS. A stir bar and 5ml of reagent water is added and the vials are capped. The soil from the third EnCore™ device is removed and immediately placed into a tared 20ml vial. The sample weight is then recorded in the weight logbook for GC or directly uploaded from the scale into the preparation batch in TALS for GCMS. The soil is then spiked with the appropriate Surrogate Standards. 10ml of Purge & Trap grade Methanol is added to the vial for Mass Spectroscopy analysis (5ml is added for GC Volatile analysis) and a cap is applied. The vial is then shaken for a period of two minutes. The vials are then stored at ≤ –7°C in an incubator specifically for 5035A volatile samples. The samples are placed in the incubator at an angle to allow the liquid room to expand during the freezing process without damaging the vials.

8.2 As an alternative method to freezing, samples can also be preserved with sodium bisulfate. At least three soil samples in Encore ™ sampling devices must be received and preservation must be performed within 48 hours of sampling and prior to storage. The soil from two of the Encore ™ devices are removed and immediately placed into tared 40ml vials containing a pre-prepared solution containing 20% sodium bisulfate. The sample weight is then recorded in the weight logbook. A stir bar is added and the vials are capped. The soil from the third EnCore™ device is removed and immediately placed into a tared 20ml vial. The sample weight is then recorded in the weight logbook for GC or directly uploaded from the scale into the preparation batch in TALS for GCMS. The soil is spiked with the appropriate Surrogate Standards. 10ml of Purge & Trap grade Methanol is added to the vial for Mass Spectroscopy analysis (5ml is added for GC Volatile analysis) and a cap is applied. The vial is then shaken for a period of two minutes. The vials are then stored at 4°C in an incubator specifically for soil volatile samples.

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass	30 grams	Cool 4 ± 2°C	14 Days [1]	SW846 3rd Edition Method 5035A Table A.1
Soils	En Core Samplers	5 grams	Freeze ≤ -7 °C	48 Hrs.[2] 14 Days[3]	SW8463 3rd Edition Method 5035A Table A.1

[1] Inclusive of preparation and analysis.
[2] Preparation only.
[3] Analysis only.

9.0 Quality Control

9.1 See appropriate method for sample preparation QC procedures.

9.2 Before the analysis of any samples, an organic-free reagent water MB will be analyzed to demonstrate that all glassware and reagents are interference free.

9.3 Methanol blanks and methanol LCS shall be prepared at the same time as methanol prepared soil samples. These methanol QC samples shall also be included in the analytical sequence with the methanol prepared soil samples.

9.4 Each analyst per methodology/instrumentation will perform an Initial Demonstration of Capability (IDOC). See determinative method for acceptable accuracy.

9.5 See appropriate determinative method for procedure to follow to demonstrate acceptable initial and continuing performance on each set of samples to be analyzed. These include the method blank, either matrix spike/matrix spike duplicate or a LCS Duplicate, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample

9.6 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[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.

9.7 Instrument QC

See appropriate determinative method for calibration and standardization

10.0 Procedure

10.1 **Data Assessment and Acceptance Criteria for Quality Control Measures**

10.1.1 See appropriate method for sample preparation QC procedures.

10.1.2 Before the analysis of any samples, an organic-free reagent water MB will be analyzed to demonstrate that all glassware and reagents are interference free. In the case of medium level analysis, an appropriate amount of methanol is also added.

10.1.3 See appropriate determinative method for procedure to follow to demonstrate acceptable initial and continuing performance on each set of samples to be analyzed. These include the method blank, either matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a Laboratory Control Sample (LCS), and the addition of surrogates to each sample and QC sample.

10.2 **Corrective Actions for Out – Of – Control Data**

10.2.1 If QC is out of range, reset samples.

10.2.2 See determinant method for QC ranges.

10.3 **Contingencies for Handling Out – Of – Control Or Unacceptable Data**

10.3.1 Inform Project Manager for client input and fill out job exception form

10.3.2 Rerun samples to confirm results.

10.3.3 Resample if client or project manager requests.

11.0 Sample Preparation

11.1 All samples are analyzed directly using the following procedure.

11.2 The sample vial containing the frozen sample, stir bar and 5ml of reagent water is removed from storage and allowed to warm to room temperature. The sample is then placed on the Archon autosampler. Without opening the vial, 5ml.of reagent water is added containing internal standards and surrogates by the autosampler. Prior to purging, the sample is heated to 40^{o}C. The sample is then purged at 40ml/min. for 11 minutes while being agitated with the stir bar. The volatile components are purged from the sample onto the trap.

11.3 The trap is rapidly heated to 245^{o}C with no flow, and then desorbed for 2 minutes with flow on to the GC column through a heated glass lined transfer line. Data is acquired. After the desorb mode, the trap is baked at 260^{o}C for 10 minutes.

11.4 If the concentration of any target analyte exceeds to the calibration range, the sample will be reanalyzed medium level. Additional data interpretation details are included in the appropriate determinative method.

11.5 Medium level method for soils is based on a solvent extraction. The 3rd Encore sample (section 8.1) is placed into a 20ml vial and weighed to the nearest 0.1g. The soil is then spiked with the appropriate Surrogate Standards. Purge and Trap grade Methanol is then added to the vial to obtain a final volume of 10ml for Mass Spectroscopy analysis (5ml for GC Volatile analysis). The weight of the sample and amount of spike added are recorded in TALS. A Methanol Blank is also prepared using the same technique with the exception of Ottawa sand being added to the vial as the matrix instead of sample. A Laboratory Control Sample is also prepared at

this time, by adding Ottawa sand into a 20ml vial and weighing it to the nearest 0.1g. The sand is then spiked with the required spiking compounds as well as the Surrogate Standards. Purge and Trap grade Methanol is then added to the vial to obtain a final volume of 10ml for Mass Spectroscopy analysis (5ml for GC Volatile analysis). The weight of the Ottawa sand and amount of spike added are recorded in the weight log book. The extracts are capped and shaken for two minutes. Matrix Spikes and Matrix Spike Duplicates are also prepared at this time by placing the 3rd Encore sample (section 8.1) into a 20ml vial and weighed to the nearest 0.1g. The soil is then spiked with the required spiking compounds as well as the appropriate Surrogate Standards. Purge and Trap grade Methanol is then added to the vial to obtain a final volume of 10ml for Mass Spectroscopy analysis (5ml for GC Volatile analysis). 1000µL aliquots of the extracts are added to 50ml of reagent water and analyzed by the appropriate determinative method.

11.6 Alternatively, the Methanol preservation can be performed in the field and the methanol preserved aliquot delivered directly to the laboratory. If this scenario is used, special sample containers and instructions must be provided to the field personnel. In addition, the weight of the pre-tarred (prior to sampling) vials must be provided and incorporated into the sample weight determination. If this option is used, the laboratory will be unable to provide any laboratory extracted QC and will allow the instruments to add the required Surrogate Standards.

11.7 A third alternative (and the least desirable) is to obtain the sample portion directly from the 4oz. jar provided for determination of dry weight. These samples will then be prepared the same as in BF-MV-005 Sections 10.5 and 10.6.

11.8 Dry weights are obtained from the same 4oz. jar used for medium level analysis. Dry weights are performed after analyses of samples are completed. 10g. of sample is weighed into a tared tin. Samples are dried overnight at 110°C.

12.0 Calibration

See appropriate determinative method for calibration and standardization

13.0 Calculations / Data Reduction

13.1 **% dry-weight** = $\frac{\text{grams of dry sample}}{\text{grams of sample}}$ X 100

14.0 Method Performance

14.1 An Initial Demonstration of proficiency will be performed by each analyst, for each, method that they are trained to perform. See determinative method for acceptable accuracy.

14.2 See appropriate determinative method

14.3 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 20.7 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.

15.0 Pollution Control

15.1 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). 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."

15.2 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."

15.3 **Waste Streams Produced by the Method**

15.3.1 The following waste streams are produced when this method is carried out.

15.3.2 Vials containing sample extracts in solvents.

15.3.3 Disposal of liquid waste is broken down into two categories: aqueous waste and solvent waste.

15.3.3.1 Aqueous waste is temporarily stored in a laboratory approved waste receptacle and labeled "A" waste.

15.3.3.2 Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste.

15.3.4 Waste receptacles are then taken to sample control, where then they are properly disposed of.

15.3.5 In the case of medium level soil extractions, the methanol is decanted off the soil into a "C" waste container. The soil is wrapped in tin foil and placed in the solid waste receptacle and then taken to sample control. Spent dry weight tins are also disposed of in this manner.

15.3.6 Glass waste, such as pipettes and vials are rinsed and disposed of in approved glass receptacles.

16.0 Waste Management

16.1 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 BF-WM-01. The following waste streams are produced when this method is carried out:

16.2 Acidic material from the auto-sampler: Waste stream must be collected in "A" waste receptacles and neutralized before discharge to a sewer system.

16.3 Methanol waste from rinses and standards: Collect in "C" waste receptacles. In the case of medium level soil extractions, the methanol is decanted off the soil and collected in the "C" receptacle. Waste receptacles are then taken to sample control where they are properly disposed of. Excess samples (acidic and non-acidic). Collect in "A" waste receptacles and neutralize samples before disposal into drain/sewer.

16.4 Excess soil sample from medium level extraction: Wrap in tin foil and place in solid waste receptacle. Soils for dry weight measurements are also disposed in this manner.

17.0 References / Cross-References

17.1 EPA, "Closed-System Purge-And-Trap and Extraction for Volatile Organics in Soil and Waste Sample", Test Methods for Evaluating Solid Waste", Volume 1B, Revision 4 1996.

17.2 USEPA CLP OLMO 4.2 / NYSDEC ASP2000 –Exhibit D – Volatiles Appendix B "Modified SW-846 Method 5035 for Volatiles in Low Level Soils

17.3 USEPA CLP OLMO 4.2A "Corrections/Modifications/Clarifications"

17.4 NYSDEC ASP 2005, July 2005

18.0 Method Modifications: None

19.0 Attachments None

20.0

21.0 Revision History

- Revision 3, dated 8 June 2012
  - Title change to accommodate the new name for the 5035A methods.
  - Section 8.1: Added the direct upload of sample weights into TALS prep batches.
  - Department Manager change, signature added.

- Revision 2, dated 12 September 2011
  - Sections 3.0, 4.0, and 9.1: VBLK name changed to MB and MSB to LCS due to new LIMS system

- Section 6.1: Software name change
- Section 11.7: Added SOP reference and corrected section references
- Department and Quality Manager change, signature added.

- Revision 1, dated 17 August 2009
  - Entered into new TestAmerica format.

Title: Thermo Scientific ICAP 6500 Analysis
Method No(s). 6010C/CLP/200.7/6010B

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

E. Scott Wagner — 10/2/2013	Kenneth Kasperek — 10/2/2013
Department Manager — Date	Technical Director — Date
Brad Prinzi — 10/2/2013	Christopher Spencer — 10/3/2013
Quality Assurance Manager — Date	Laboratory Director — Date

Facility Distribution No. _____ **Distributed To:_____**

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		

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 occur. Current IDLs and MDLs are maintained in the laboratory LIMS.

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% HNO_3 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.
- 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 LLICV – 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.

3.14 LLCCV – 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. MB- Method Blank – A blank sample that is taken through each step of the analytical procedure, including the digestion procedure if it is used.

3.17 Calibration Blank - A blank solution containing 6% HNO_3 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 $M\Omega cm^{-1}$

3.21 LLQC- Lower limit of quanitation- Digested and analyzed to confirm the lowest quanitation limit

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 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.

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. Spiking a sample consists of adding a specified amount of four separate spike solutions to the unknown sample. Each spike solution contains various elements of interest.

4.5.3 The five 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.

ANALYTE	TRUE VALUE
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% HNO_3 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.

- **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.

ANALYTE	TRUE VALUE
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% HNO_3 in water. All weights are traceable to NIST traceable weights.

- **Spike 3 (prepared by lab)**

ANALYTE	TRUE VALUE
Silver	10 µg/mL

Matrix 2% HNO_3 in water. See 7.10 for preparation.

- **Spike 4 (prepared by lab)**

ANALYTE	TRUE VALUE
Tin	40 µg/mL

Matrix: 5% HNO_3 in water. See 7.11 for preparation.

- **Spike 5 (prepared by lab)**

ANALYTE	TRUE VALUE
Silicon	2000 µg/mL

Matrix: 5% HNO_3 in water. See 7.12 for preparation.

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 and Spike 5 to 9.75 mL of sample. Mix thoroughly and analyze.

4.5.4 The three spike solutions for CLP samples are:

- **CLP-1 Made by ULTRA SCIENTIFIC (ICUS-987)**

ANALYTE	TRUE VALUE
Aluminum	1000 µg/mL
Barium	1000 µg/mL
Beryllium	25.0 µg/mL
Chromium	100.0 µg/mL
Cobalt	250.0 µg/mL
Copper	125.0 µg/mL
Iron	500.0 µg/mL
Manganese	250.0 µg/mL
Nickel	250.0 µg/mL
Silver	25.0 µg/mL
Vanadium	250.0 µg/mL
Zinc	250.0 µg/mL

- **CLP-2 Made by ULTRA SCIENTIFIC (ICUS-988)**

ANALYTE	TRUE VALUE
Antimony	50.0 µg/mL

- **CLP-3 Made by ULTRA SCIENTIFIC (ICUS-989)**

ANALYTE	TRUE VALUE
Arsenic	20.0 µg/mL
Cadmium	25.0 µg/mL
Thallium	25.0 µg/mL
Selenium	25.0 µg/mL
Lead	10.0 µg/mL

Refer to sample preparation SOPs for the preparation of matrix spikes for CLP samples. The spike recovery criteria may be found in section 9.8.1.

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% HNO_3, 5% HCl by volume. Preserved metals samples contain 1-2% HNO_3 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.**

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.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

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.

- Spare parts for the ICAP:
  - nebulizers
  - torches
  - spray chambers
  - rotors
  - stators
  - fast switches
  - duo radial plasma view window

- White/White or Gray/Gray pump tubing (drain)
- Orange/Green pump tubing (IS)
- Black/Black pump tubing (carrier)
- 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

- 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 $M\Omega cm^{-1}$ 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% HNO_3

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 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. 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% HNO_3 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 1200 mL concentrated HNO_3 and 1000 mL concentrated HCl to a 20 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.

7.10 Spike 3 (or Ag Spike), containing 10 μg/mL Ag, is prepared by adding 1.0 mL of 1,000 μg/mL Ag stock standard to a 100 mL volumetric flask half filled with 2% HNO_3 Blank Solution. Bring to the final volume with 2% HNO_3 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 4.0 mL of 1,000 ug/ml Sn stock standard to a 100 mL volumetric flask filled with blank solution. Bring up the final volume with Blank solution. This spike is used for the post-spike.

7.12 Spike 5 (or Si Spike), containing 2000 μg/mL Sn, is prepared by adding 20.0 mL of 10,000 ug/mL Si standard to a 100 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 The following calibration standards and quality control standards are prepared in the laboratory from ULTRA SCIENTIFIC 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)

7.13.1 IC2 is prepared by adding 20 mL of IC4 (Section 7.13.3) 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 IC2.

7.13.2 IC3 is prepared by adding 100 mL of IC4 (Section 7.13.3) 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.

7.13.3 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 μg/mL Ag, Sn; and 2.5 mL 10,000 μg/mL Si 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.

7.13.4 The ICSA is prepared by adding 50.0 mL of ICSA stock solution (ICM-441) 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.

7.13.5 The ICSAB is prepared by adding 50.0 mL of ICSAB stock solution (ICUS-3482) and 0.1 mL 1,000 μg/mL Ag stock standard 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.

7.13.6 The Low Level Verification standard (CRI/ICVL/CCVL) is prepared by adding 50 mL of stock standard ICUS-3099 to a 500 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.

7.13.7 Internal Standard: The IS is prepared by adding 0.5 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.

7.14 The following quality control standards are prepared in the laboratory from HIGH PURITY stock standards:

7.14.1 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, to a 1000 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.

7.14.2 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.

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 $\pm$ 2°C	180 Days	N/A

[1] Inclusive 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 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 (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) – 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 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)[4]	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) 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.
[3] A Matrix Duplicate may be used in place of a Matrix Spike Duplicate for some methods

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 +/- 10% of the true value for CLP and method 6010, or within +/- 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 - 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.

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.

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 Quanitations (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, LLICV 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 can not be used for the out of control analyte(s).

9.4.4 If a CCV, LLCCV, 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 falls outside the control limits of 70-130% (50-150% for Sb, Pb, Tl), the CRI must be re-analyzed for the outlying analytes. The same is true if the CRI falls outside the control limits of 50-150% for 6010B and 200.7, and if the LLICV/LLCCV 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 ICB, 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 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 glass 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.

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.

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 or 6010B analytical run is typed in the following format:

ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
↑
10 samples
↓
CCV
CCB

↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB

CRI	**Client specific (QAPPs)**
ICSA	↓
ICSAB	↓
CCV	↓
CCB	↓

Run a CCV and CCB after every 10 samples and at the end of the analytical run. Run the CRI, 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 these QC samples are required for a particular protocol. For example, the ending CRI, ICSA and ICSAB are not required by SW-864 (6010B) and 40 CFR protocol. Therefore, a particular run may not include ending CRI, 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 Non-CLP 6010C analytical run is typed in the following format:

ICV
LLICV
ICB
ICSA
ICSAB
CCV
CCB
LLCCV
↑
10 samples
↓
CCV
CCB
LLCCV
↑

10 samples
↓
CCV
CCB_
LLCCV
↑
10 samples
↓
CCV
CCB
LLCCV
↑
10 samples
↓
CCV
CCB
LLCCV
↑
10 samples
↓
CCV
CCB
LLCCV

ICSA	**Client Specific**
ICSAB	↓
CCV	↓
CCB	↓
LLCCV	

Run a CCV, CCB, and LLCCV after every 10 samples and at the end of the analytical run. Run ICSA and ICSAB at the beginning and end of the analytical run if specified by the client.

10.3.2.3 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
↑
7samples
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
  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"
- Choose "_All Elements_" on left menu of auto-peak screen.
- 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 9.75 mL of sample:

50 µL - Spike 1 (Section 4.5.3)
50 µL - Spike 2 (Section 4.5.3)
50 µL – Spike 3 (Section 4.5.3)
50 µL – Spike 4 (Section 4.5.3)
50 µL – Spike 5 (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.

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 button that will establish communication between the autosampler and the instrument.
- 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. Thee 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, LLCCV, LLICV, 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.

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).

$$\%\,RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} X\ 100$$

11.1.1 The formula for calculating the relative percent difference is:Where,
RPD = relative percent difference
D_1 = first sample value
D_2 = 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:

$$\% \ Recovery = \frac{S_2 - S_1}{SA} \ X \ 100$$

Where, S_2 = the post spiked sample reading
S_1 = 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.

$$\% Recovery = \frac{2.289 - .250}{2.000} X \ 100$$

$$\% \ Recovery = 102.0\%$$

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.

$$C_x = \frac{(0.792)(0.05)(200.0)}{1.512 - 0.792)(10.0)}$$

$$C_x = \frac{7.92}{7.20}$$

$$C_x = 1.10 ppm$$

12.0 Method Performance

This SOP is applicable to digested sample matrices and soluble water samples.
Ok

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.

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 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 HNO_3 waste in an "AN" waste container.

15.0 References / Cross-References

15.1 Method 6010B and Method 6010C Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996.

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.

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 BS, 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/LLCCV/LLICV 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 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
- 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.
  - 10.3.5.1: Added Spike 5
  - 10.3.7: Changed Excel to Open Office.org Calc
  - 17.15 and 17.16: Added Interfering element tables.

- **Revision 3, Dated June 03, 2011**
  - 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.
  - Section 1.0: Reorganized and reworded Scope and Application Section
    - 1.3.2 Analyte table added, including lithium, strontium and silicon.
  - Section 3.0: Removed section 3.21.
  - Section 4.0: Removed reference to lithium nitrate buffer – no longer used
  - 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.
  - Section 5.0: Section reorganized and renumbered, removed section 5.3.
  - Section 6.0: Section changed to pertain to new instrumentation.
  - 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

- Section 7.9: Spike 5 added (for Si).
- Inserted Section 7.12 for Spike 5 preparation – subsequent section 7.x incremented accordingly.
- 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.
- Section 9.0: Section reorganized and updated to reflect TestAmerica Method SOP Template.
    - Added requirements for 6010C.
        - Section 9.2 correlation requirement changed from .995 to .998.
        - Added lab procedure for diluting a negative value in section 9.4.12.
- 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.
- Section 12.0: Replaced references to Trace #1 and Trace #2 with ICAP 1 and ICAP 2, included 6010C requirements.
- Section 15.0: Added reference to ICAP 6500 Analyzer Operator's Manual.
- Section 17.0: Removed 17.11 through 17.15 – old ICPs or redundant information.
- Section 17.14: example certificates of analysis updated for new ICUS standards.
- 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
- Throughout SOP: changed procedures and nomenclature to pertain to new instrumentation.
    - ICAP 1 and ICAP 2 replaced Trace #1 and Trace #2

- Changed Quality Manager, signature changed.

- **Revision 2, Dated January 18, 2010**
  - Removed 3.8 ICL-HCV-The highest calibration standard re-run directly after calibrating the instrument.
  - Section 4.5.1 Removed "such that the analyte in the diluted sample is at least a factor of 10 above the IDL"
  - 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."
  - Section 6.1 Changed STL: Buffalo to TestAmerica Buffalo
  - Section 7.6 Removed standard logbook and added in Element
  - 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 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."
  - Remove 9.3 "ICL-The ICL is the highest calibration standard that is analyzed after the instrument is calibrated."
  - Section 9.4 Removed "It is analyzed after the ICL."
  - Section 9.10 Changed LFB to BS. Changed Laboratory Fortified Blank to Blank Spike.
  - Section 9.11 Changed LCS to SRM. Changed Laboratory Control Sample to Sample Reference Material.
  - Section 9.21.5 Changed LFB and LCS to BS and SRM.
  - Section 9.21.9 Changed LCS to BS.
  - Section 10.3.1 Changed STL Buffalo to TestAmerica Buffalo
  - Section 10.3.6.2 Changed "a piece of parafilm" with the lid.
  - Section 10.4.1 Removed HCV
  - Section 10.4.2 Removed HCV
  - Section 16.0 Added method modification
  - Removed Tables 17.19 and 17.20
  - Table 17.4 Added nm next to wavelength
  - Section 2.3 Removed AFCEE reference
  - Section 10.4.1 removed AFCEE references
  - Section 12.4 removed USACE reference
  - Section 7.6 changed 'standards logbook' to LIMS
  - Section 9.1 changed 'stndards logbook' to LIMS
  - Section 10.3.6.5 rewords the procedure to clean the torch.
  - Section 17.7 removed ICUS-573 standard. No longer used

- **Revision 1, Dated July 07, 2009**
  - New LIMS nomenclature changes:
  - BLANK became CAL1
  - Std.1 became CAL2
  - Std.2 became CAL3
  - Std.3 became CAL4
  - Std.3 VER became HCV
  - CRI became LCV
  - ICSA became IFA
  - ICSAB became IFB
  - MBLK became BLK
  - LCS became SRM
  - LFB became BS
  - SD became MSD
  - 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."
  - 10.4 Typing an Analytical Run: "SEQ-"added before all calibration and QC standards, names updated.
  - 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."
  - 17.0 Example of a Data Review Summary Form for Metals replaced with an Example of a Bench Sheet for Metals.
  - Metals Department manager change, signature.

- **Revision 0, Dated June 09, 2008**
  - Integration for TestAmerica operations
  - Quality Manager change, signature

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		

17.2 Approximate Water Detection Limits for the ICAP 6500 Analyzers.

Element	Estimated IDL (mg/L)	Estimated MDL (mg/L)	Lab PQL (mg/L)
Al	0.0563	0.060	0.2
Sb	0.0038	0.00679	0.02
As	0.0035	0.0055	0.01
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	0.001
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	0.005
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	0.015
Na	0.0920	0.324	1.0
Ag	0.00079	0.0017	0.003
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
Mn	0.00018	0.0003	0.003

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
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
Sn	189.989
Sr	407.771
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	500	500
Sb	50	50
As	20	20
Ba	10	10
Be	5	5
B	50	50
Cd	20	20
Ca	1000	1000
Cr	40	40
Co	20	20
Cu	25	25
Fe	500	500
Pb	60	60
Li	50	50
Mg	500	500
Mo	10	10
Ni	25	25
K	600	600
Se	40	40
Na	1000	1000
Ag	2	2
Si	1000	1000
Sr	10	10
Tl	40	40
V	50	50
Zn	10	10
Sn	5	5
Ti	10	10
Mn	20	15

17.6 Concentration of each analyte for LCS, SRM, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike:

Element	LCS, Post-Digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (mg/L)	Soil Post-Digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Representative Soil LCS (mg/kg) changes per lot	CLP Matrix Spike Soil (mg/kg)
Al	10	2.00	200	16300	-
Sb	0.20	0.100	40	117	20
As	0.20	0.040	40	138	8
Ba	0.20	2.00	40	269	400
Be	0.20	0.050	40	157	10
B	0.20	-	40	90	-
Cd	0.20	0.050	40	71	10
Ca	10	-	200	9660	-
Cr	0.20	0.200	40	105	40
Co	0.20	0.500	40	142	100
Cu	0.20	0.250	40	110	50
Fe	10	1.00	200	19100	200
Pb	0.20	0.020	40	144	4
Li	0.20	-	40	-	-
Mg	10	-	200	4410	-
Mo	0.20	-	40	90.4	-
Ni	0.20	0.500	40	130	100
K	10	-	200	5000	-
Se	0.20	0.050	40	200	10
Na	10.0	-	200	653	-
Ag	0.20	0.050	40	45.1	10
Si	10	-	200	-	-
Sr	0.20	-	40	246	-
Tl	0.20	0.050	40	161	10
V	0.20	0.500	40	67	100
Zn	0.20	0.500	40	268	100
Sn	0.20	-	40	160	-

Element	LCS, Post-Digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (mg/L)	Soil Post-Digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Representative Soil LCS (mg/kg) changes per lot	CLP Matrix Spike Soil (mg/kg)
Ti	0.20	-	40	447	-
Mn	0.20	0.500	40	539	100

17.7 Reagents and Stock Solutions which are Purchased as Starting Materials for Preparation of Trace Standards.

From ULTRA SCIENTIFIC:

ICM-441
ICUS-3098 (formerly 576)
ICUS-3099 (formerly 1932)
ICUS-3100 (formerly 919)

CLP-1
CLP-2
CLP-3

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 ug/mL Y *
10,000 ug/mL Zn

Certificates of Analysis are attached for the custom blend standards listed as ICUS-(…) above.

From HIGH PURITY:

1,000 µg/mL Ag	CAL STD #2-R Solution A
1,000 µg/mL Sn	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 HNO_3 (Trace Metals Grade)

Table 17.8 Concentrations of Calibration Standards: (in mg/L)

Element	IC2	IC3	IC4
Al	5.0	25.0	50.0
Sb	0. 1	0.5	1.0
As	0. 1	0.5	1.0
Ba	0. 1	0.5	1.0
Be	0. 1	0.5	1.0
Cd	0. 1	0.5	1.0
Ca	5.0	25.0	50.0
Cr	0.1	0.5	1.0
Co	0.1	0.5	1.0
Cu	0.1	0.5	1.0
Fe	5.0	25.0	50.0
Li	0.1	0.5	1.0
Mg	5.0	25.0	50.0
Mn	0.1	0.5	1.0
Ni	0.1	0.5	1.0
Ag	0.1	0.5	1.0
Si	5.0	25.0	50.0
Sr	0.1	0.5	1.0
Tl	0.1	0.5	1.0
Zn	0.1	0.5	1.0
V	0.1	0.5	1.0
B	0.1	0.5	1.0
Mo	0.1	0.5	1.0
Ti	0.1	0.5	1.0
Sn	0.1	0.5	1.0
Se	0.1	0.5	1.0
Na	5.0	25.0	50.0

K	5.0	25.0	50.0
Pb	0.1	0.5	1.0

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	-

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

Element	CCV	ICV
Mn	0.5	0.375

Table 17.11 Contract Required Detection Limits (CRDL)

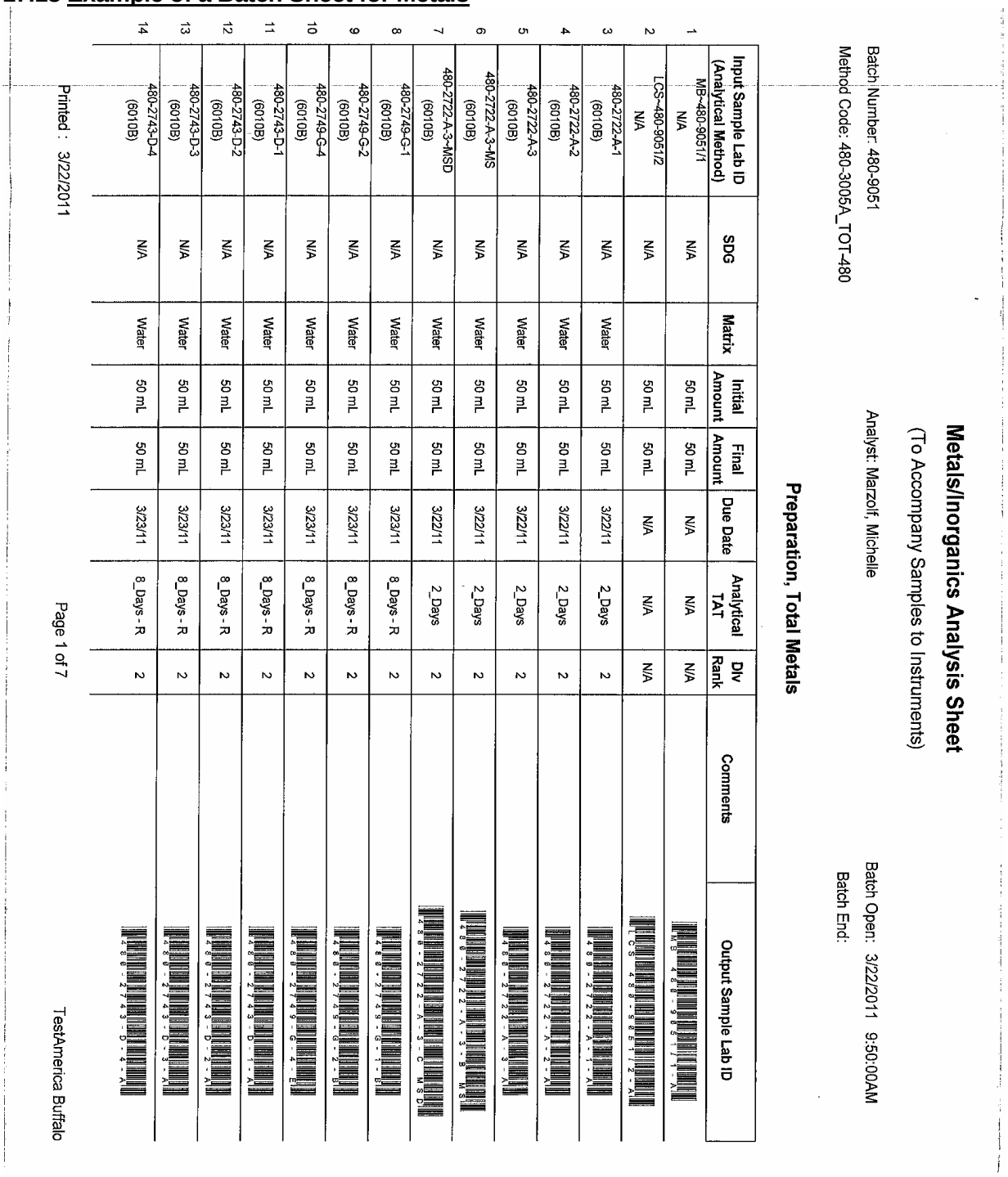
Analyte	CRDL (4.0) (ng/mL)	CRDL (5.0) (ng/mL)
Aluminum	200	200
Antimony	60	5
Arsenic	10	5
Barium	200	20
Beryllium	5	1
Cadmium	5	2
Calcium	5000	5000
Chromium	10	5
Cobalt	50	5
Copper	25	5
Iron	100	100
Lead	3	3
Magnesium	5000	5000
Manganese	15	10
Mercury	0.2	0.1
Nickel	40	20
Potassium	5000	5000
Selenium	5	5
Silver	10	5
Sodium	5000	5000
Thallium	10	5
Vanadium	50	10
Zinc	20	10

17.12 Element Concentrations in the CRI / LLICV / LLCCV Stock and Working Standards

Analyte	CRI / LLICV / LLCCV Stock Std. ICUS-1932	CRI / LLICV / LLCCV Working Std.
Aluminum	2.0	0.2
Antimony	0.2	0.02
Arsenic	0.1	0.01
Barium	0.02	0.002
Beryllium	0.02	0.002
Boron	0.2	0.02
Cadmium	0.01	0.001
Calcium	5.0	0.5
Chromium	0.04	0.004
Cobalt	0.04	0.004
Copper	0.1	0.01
Iron	0.5	0.05
Lead	0.05	0.005
Lithium	0.3	0.03
Magnesium	2.0	0.2
Manganese	0.03	0.003
Molybdenum	0.1	0.01
Nickel	0.1	0.01
Potassium	5.0	0.5
Selenium	0.15	0.015
Silver	0.03	0.003
Sodium	10.0	1.0
Silicon	5.0	0.5
Strontium	0.05	0.005
Thallium	0.2	0.02

Tin	0.1	0.01
Titanium	.05	0.005
Vanadium	0.05	0.005
Zinc	0.1	0.01

17.13 Example of a Batch Sheet for Metals



Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 480-9051
Method Code: 480-3005A_TOT-480

Analyst: Marzolf, Michelle

Batch Open: 3/22/2011 9:50:00AM
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 (6010B)	N/A		50 mL	50 mL	N/A	N/A	N/A		MB 480-9051/1-A
2	LCS-480-9051/2 (6010B)	N/A		50 mL	50 mL	N/A	N/A	N/A		LCS 480-9051/2-A
3	480-2722-A-1 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		480-2722-A-1-A
4	480-2722-A-2 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		480-2722-A-2-A
5	480-2722-A-3 (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		480-2722-A-3-A
6	480-2722-A-3-MS (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		480-2722-A-3-B MS
7	480-2722-A-3-MSD (6010B)	N/A	Water	50 mL	50 mL	3/22/11	2_Days	2		480-2722-A-3-C MSD
8	480-2749-G-1 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2749-G-1-D
9	480-2749-G-2 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2749-G-2-B
10	480-2749-G-4 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2749-G-4-E
11	480-2743-D-1 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2743-D-1-A
12	480-2743-D-2 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2743-D-2-A
13	480-2743-D-3 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2743-D-3-A
14	480-2743-D-4 (6010B)	N/A	Water	50 mL	50 mL	3/23/11	8_Days - R	2		480-2743-D-4-A

Printed : 3/22/2011

Page 1 of 7

TestAmerica Buffalo

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		

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 B1 CCV+CCB
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

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, Tl, 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, Tl, V, Zn	1:5 K+Na CCB↑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↑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	1

Analytes that are not being reported with be displayed in [...] brackets. Analytes that are not being reported but are on the spike list with be displayed in (...) parentheses.

I 203221 1B-4

I 103231 1A-2

MB B
LCS 1
2724-1 1:5 B, K, Na
2724-2 1:5 K, Na
2326-11 B
2326-12 1

17.14 Certificates of Analysis for Custom Blend Standards

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

RT00731
NEED: 1/14/10
DAN

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 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

ACCREDITED
ISO 17025:2005
Accredited
A2LA
Cert. No. 0851.01

ISO 9001:2000
Registered
TUV USA, Inc.
Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

See Reverse For Additional Information

William J. Leahy
Quality Assurance Manager

Facility Distribution No. _____ Distributed To:_____

RT14762 R: 11/26/10 AMH
E: 9/30/12

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

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

ACCREDITED
ISO 17025:2005
Accredited
A2LA
Cert. No. 0851-01

ISO 9001:2008
Registered
TUV USA, Inc.
Cert. No. 08-1009

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 295-2330
www.ultrasci.com

William J. Leary
Quality Assurance Manager

Facility Distribution No. _____ **Distributed To:_____**

Spike 2

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

family 574

Catalog Number: ICUS-3097
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

ACCREDITED
ISO 17025:2005
Accredited
A2LA
Cert. No. 0851.01

ISO 9001:2000
Registered
TUV USA, Inc.
Cert. No. 08-1004

William J. Leary
Quality Assurance Manager

250 Smith Street, North Kingstown, RI 02852 USA
Ph: 401-294-9400 * Fax: 401-295-2330
www.ultrasci.com

Facility Distribution No. _____ **Distributed To:_____**

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

formerly 576

Catalog Number: ICUS-3098
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	5006	±	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

ACCREDITED
ISO 17025:2005
Accredited
A2LA
Cert. No. 0851.01

ISO 9001:2000
Registered
TUV USA, Inc.
Cert. No. 06-1004

William J. Leary
Quality Assurance Manager

250 Smith Street, North Kingstown, RI 02852 USA
Ph: 401-294-9400 * Fax: 401-295-2330
www.ultrasci.com

Facility Distribution No. _____ **Distributed To:_____**

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-3099 formerly 1392
Lot Number: M00385
Job Number: J00012949
Lot Issue Date: 04/14/2011
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
aluminum	2.000	± 0.010	µg/mL	gravimetric
antimony	0.2000	± 0.0010	µg/mL	gravimetric
arsenic	0.1000	± 0.0005	µg/mL	gravimetric
barium	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/NCSL Z-540-1 and ISO 9001

ACCREDITED

ISO 17025:2005 Accredited A2LA Cert. No. 0851-01

ISO 9001:2008 Registered TUV USA, Inc. Cert. No. 09-1009

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 295-2330
www.ultrasci.com

William J. Leary
Quality Assurance Manager

Facility

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

formerly 919

Catalog Number: ICUS-3100
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	6.000	±	0.030	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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ISO 17025:2005 Accredited A2LA Cert. No. 0851.01

ISO 9001:2000 Registered TUV USA, Inc. Cert. No. 06-1004

William J. Leary
Quality Assurance Manager

250 Smith Street, North Kingstown, RI 02852 USA
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www.ultrasci.com

Facility Distribution No. _____ **Distributed To:_____**

PECO 10/15/10 PT12812

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

SM-606-044 (CAL STD #2RR)
Solution A
Lot # 1027723

Source	Source Purity	Matrix	Standard Concentration
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 0 6 2011
MSDS ATTACHED

RECD 10/15/10 RTT2813

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

SM-606-044 (CAL STD #2RR)
Solution B
Lot # 1027724

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.96+ %	HNO_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 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 0 6 2011
MSDS ATTACHED

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

RT10384
RECD: 8/24/10
ATW

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 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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William J. Leary
Quality Assurance Manager

Facility Distribution No. _____ **Distributed To:_____**

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,Tl,V
Co	Ni Pb Tl	V	Al,Cd,Tl, Be
Cr	As Sb V Zn	Mo	B Co Pb
Fe	Cd Cr,Pb,V		
Mn	V		

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	Tl	Ni
Mn	Tl	V	Al
	V		Ag
Mo	As		Be
	B		Cd
	Co		Cu
	Pb		Tl
	Sb		

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

BF-QA-SOPICF-017
Rev.0 2/27/13

TestAmerica Buffalo
SOP Interim Change Form

SOP Number: BF-ME-011	**IC Number:** 1025
SOP Title: MERCURY PREPARATION AND ANALYSIS	
SOP Sections Affected by Change: 9.1.6	

Reason for Addition or Change:

ADD:
MATRIX SPIKE
MATRIX SPIKE DUPLICATE

80 - 120% RECOVERY UPDATE 4

Submitted By: SCOTT WAGNER Date: 8/20/13

APPROVED BY:

Department Supervisor: [signature] Date: 8/20/13

QA Manager: [signature] Date: 9/4/13

Laboratory Manager: [signature] Date: 9/4/13

Laboratory Director: [signature] Date: 9/4/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

BF-QA-SOPICF-017
Rev.0 2/27/13

TestAmerica Buffalo
SOP Interim Change Form

SOP Number: BF-ME-011	**IC Number:** 1026
SOP Title: MERCURY PREPARATION AND ANALYSIS	
SOP Sections Affected by Change: 9.2.8 TABLE CONTROL LIMIT	
Reason for Addition or Change: TO ADD: CCV 90 - 110% RECOVERY - UPDATE 4	
Submitted By: SCOTT WAGNER	Date: 8/20/13

APPROVED BY:

Department Supervisor: Scott Wagner Date: 8/20/13

QA Manager: [signature] Date: 9/4/13

Laboratory Manager: Jennifer Spence Date: 8/20/13

Laboratory Director: [signature] Date: 9/4/13

Title: Mercury Preparation and Analysis [Methods 245.1, 7470A, 7471A, 7471B]

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

E. Scott Wagner — 4/25/2013	Jennifer Pierce — 4/25/2013
E. Scott Wagner — Date	Jennifer Pierce — Date
Department Manager	Operations Manager
Brad Prinzi — 4/25/2013	Christopher Spencer — 4/25/2013
Brad Prinzi — Date	Christopher Spencer — Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. _____ Distributed
To:_____

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 ($K_2S_2O_8$) (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 ($NH_2OH \bullet HCl$) 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 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 material safety data sheets (MSDS's) are made available to all personnel involved in these analyses. All MSDS's 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 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
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. Temperature 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 µg/L to 10.0 µg/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: (DI H_2O); 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 corro*sive.

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 ($K_2S_2O_8$): Prepare by dissolving 100 g of $K_2S_2O_8$ 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 / NH_2OH•HCl): (*abbrev. HyHy*); Prepare by dissolving 240 g of NaCl and 240 g of NH_2OH-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 $K_2S_2O_8$ and 40 mL of Hydroxlyamine 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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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℃ 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

[1] Inclusive 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 400 mL of EBLK to a 500 mL bottle and spike with 2.0 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:

$$4\ \mu g/L \times 0.050\ L \div 2.170\ \mu g/g = 0.09216\ g$$

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 Calibration Verification (CRA): 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 must be analyzed following the ICV and ICB at the beginning of any an analytical sequence. (Note: Many client QAPPs require that an additional CRA be analyzed at the end of each analytical sequence. Unless there is a specific reason not to, is a good practice to analyze an CRA at the end of each analytical sequence.) Formerly referred to as LCV.

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	0.2 ppb	Beginning of each analytical sequence. End of each analytical sequence for update 4.	50-150% recovery, 70-130% for MCP/RCP 70%-130% recovery – 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 / 5

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 ($KMnO_4$)
- 2.0 mL Potassium Persulfate ($K_2S_2O_8$)

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 2.5 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 (MnO_2) may remain on the bottom or sides of the digestion cup. It is of no concern, and may dissolve over time.

10.1.6 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 (SiO_2). Add approximately 5-10 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 (HNO_3). **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:
- 5-10 mL Reagent Water
- 10-15 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 2.5 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 Repetitons 1-3) to generate the calibration curve. In addition to the control limits specified above, a correlation coefficient (rho) 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.

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. Auto-sampler 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). 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 C4, C3, C1 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	
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	C4 C3 C1
14			1.0000	1.0000	

The above table would result in a run sequence as follows:

ICV
ICB
CRA
CCV
CCB
↑
10 samples (with the given ID#s)
↓
CCV
CCB
↑
3 samples
↓
CRA
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, 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

$$\text{ICV / CCV / CRA / LCS \%Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS / MSD \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Wet-Weight Basis

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 $\times$ 0.001 = kg)

11.4 Percent Solids

To report percent solids in solid samples, calculate as follows:

% 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 falls out of acceptance limits, discontinue the analysis to correct the problem, 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 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 MSDS 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	*Environmental Express* 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 7, dated 25 April, 2013
  - Section 6.2.2 added reference to include both beginning and ending temperature measurements.
  - Section 10.3.1.10 changed sample ID to include use for sample ID's less than 10 characters.
  - Section 10.3.1.10 removed reference to S and P macro Code combination for determination of spike recoveries (no longer used).
  - Section 12.1 removed reference to yearly MDL determination
  - Section 9.1.5 addition of MCP/RCP assignment of MS/MSD
  - Section 9.1.6 added MCP/RCP to LCSD and MS/MSD criteria
  - Table 9.2.8 added CRA criteria of 70-130% for MCP/RCP
  - Quality Manager updated, signature added

- Revision 6, dated March 28, 2012
    - Section 10.2.2 added reference to include soil digestion to match the matrix of the digested samples.
    - Section 10.2.2.2 addition of 10.1.6 to reference the soil digestion procedure.
- Revision 5, dated January 19, 2012
    - Changed Standard Reference Material (SRM) to Laboratory Control Sample Standard Reference Material (LCSSRM). Throughout.
    - Changed LCV to CRA throughout.
    - Changed Method Blank (BLK) to Method Blank (MB) throughout.
    - Changed Matrix Duplicate (MD) to Matrix Duplicate (DU) throughout.
    - Changed Serial Dilution (SRD) to Serial Dilution (SD).
    - 9.1.1.3 Changed TCLP extraction blank TALS ID from BLK to LB.
    - 6.2.7 Changed scale model from Mettler AE200 to Denver P-214.
    - 10.2.3.3, 10.3.1.4 removed references to the rinse bath.
    - 10.3.1.10 Due to character limitations batch sample IDs are entered into extended ID instead of sample ID.
    - Updated Example Autosampler Table to reflect changes in section 10.3.1.10.
    - Deleted S P out of Example Autosampler Table.
    - 10.3.2.2 H-Drive folder changed from Ward/Sdgs/Instdata/Mercury to H:\Mercury\Lims.
- Revision 4, dated 15 February 2011
    - Replaces previous SOP BF-ME-011, revision 3
    - Added reference to Method 7471B in Section 1.2 and Section 15.0
    - Replaced all references to Blank Spikes (BS) to Laboratory Control Samples (LCS).
    - 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
    - Replaces previous SOP BF-ME-011, revision 2
    - Spelling, Grammar, & Formatting corrections
    - Section 4.1.9 Added specific guideline for sample reanalysis to check for carryover from high level samples
    - Section 4.2 & 4.3 Additional detail provided concerning various chemical vs. physical interferences
    - Section 9.1.2.3 Removed references to AFCEE and USACE
    - Sections 6.26, 6.27, 6.2.10, 10.2.3.7 Added missing cross-references to other relevant SOPs
    - Attachment 4 - Title renamed for clarity
- Revision 2, dated 02 September 2009
    - Replaces previous SOP BF-ME-011, revision 1

- 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.
- 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).
- 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.
- 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%
- 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
- Added details concerning data file and autosampler file naming conventions
- Added details concerning sample preparation cup and analysis test tube numbering system
- 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.
- Reformatted sample preparation section to separate Aqueous from Solid digestion steps
- 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
- Added Sample Calibration Page Attachment
- Updated Example batch attachments from AIMS to ELEMENT batches
- Changed potassium permanganate added to soil samples from 10 mL to 10-15mL.

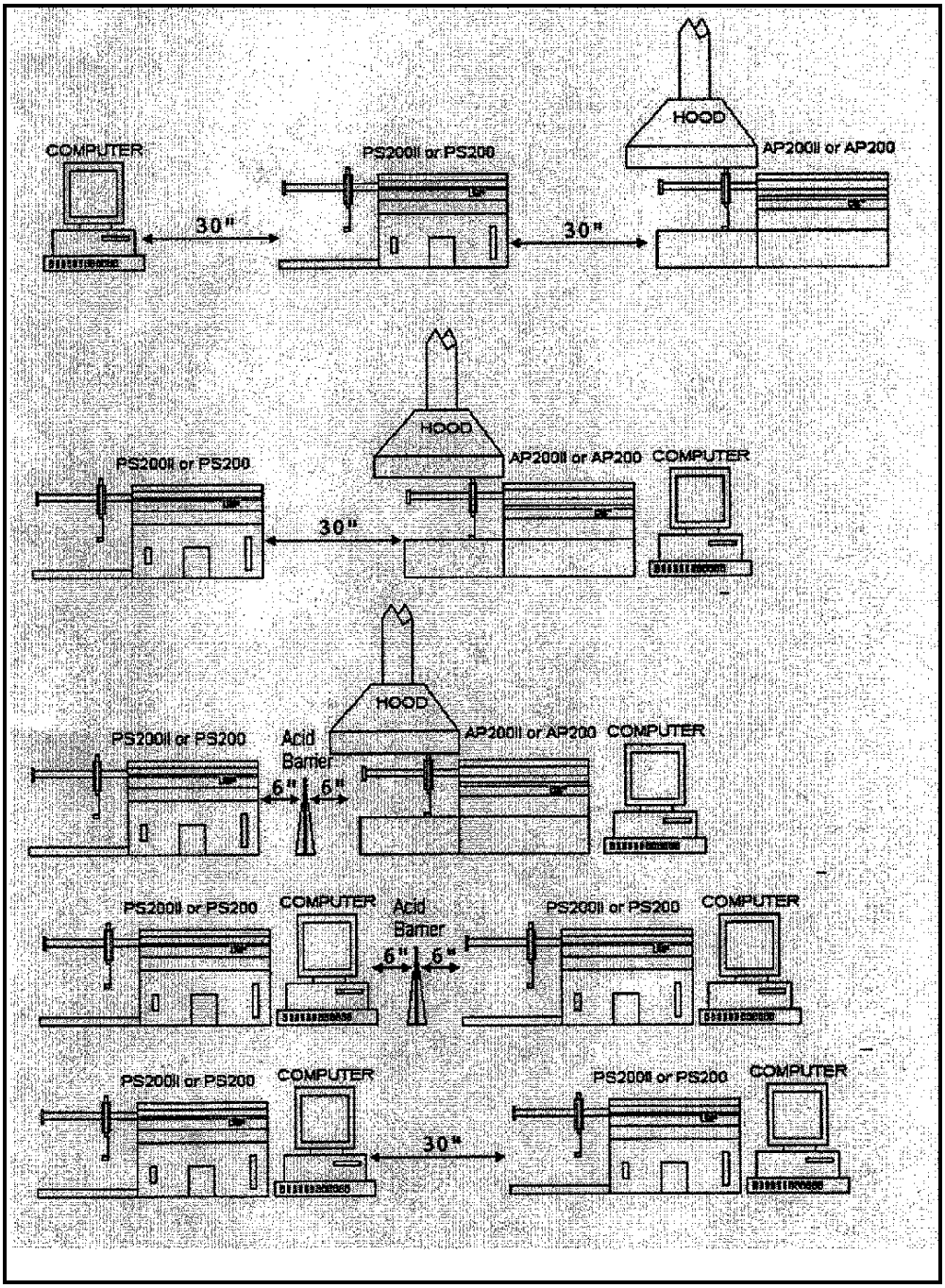
- Revision 1, dated 15 April 2008
    - Replaces previous SOP BF-ME-011, revision 0
    - Sections 10.3.7, 10.3.8, 10.3.9 and 10.4. Edited for improved clarity regarding stock standard, intermediate standard, and working standard preparation; and to correct copying errors introduced in transitioning SOP formats from AME-MERCURY-50, rev.7 to BF-ME-011, rev.0.
    - Section 12.7 and 12.8. Correct 100 ppb to Hg#2 instead of Hg#1
    - 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.
    - 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
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

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	480-1516-G-1 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2		
11	480-1516-G-2 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2		
12	480-1516-G-3 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2		
13	480-1569-F-1 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2		
14	480-1569-F-2 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2		

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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	480-1569-F-3 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2	
16	480-1569-F-4 (7470A)	N/A	Filtrate	30 mL	50 mL	2/16/11	10_Days - R	2	
17	480-1569-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	

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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	039576
Hydroxylamine Hydrochloride Lot	039590
NaCL Lot #	
Repittetor Volume Check	
SOP Number	

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Attachment 2

Sample Water Digestion Batch Bench Sheet (Page 4of 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

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#:

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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
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

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		+0.6250 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	+0.6609 g	50 mL	2/11/11	8_Days - R	4		
4	480-1553-C-1 (7471A)	N/A	Solid	+0.6374 g	50 mL	2/16/11	8_Days - R	2		
5	480-1553-C-1~SD (7471A)	N/A	Soli					2		
6	480-1553-C-1~MS (7471A)	N/A	Soli					2		
7	480-1553-C-1~MSD (7471A)	N/A	Solid	+0.6230 g	50 mL	2/16/11	8_Days - R	2		
8	480-1553-B-2 (7471A)	N/A	Solid	+0.6139 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		

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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 Number	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	

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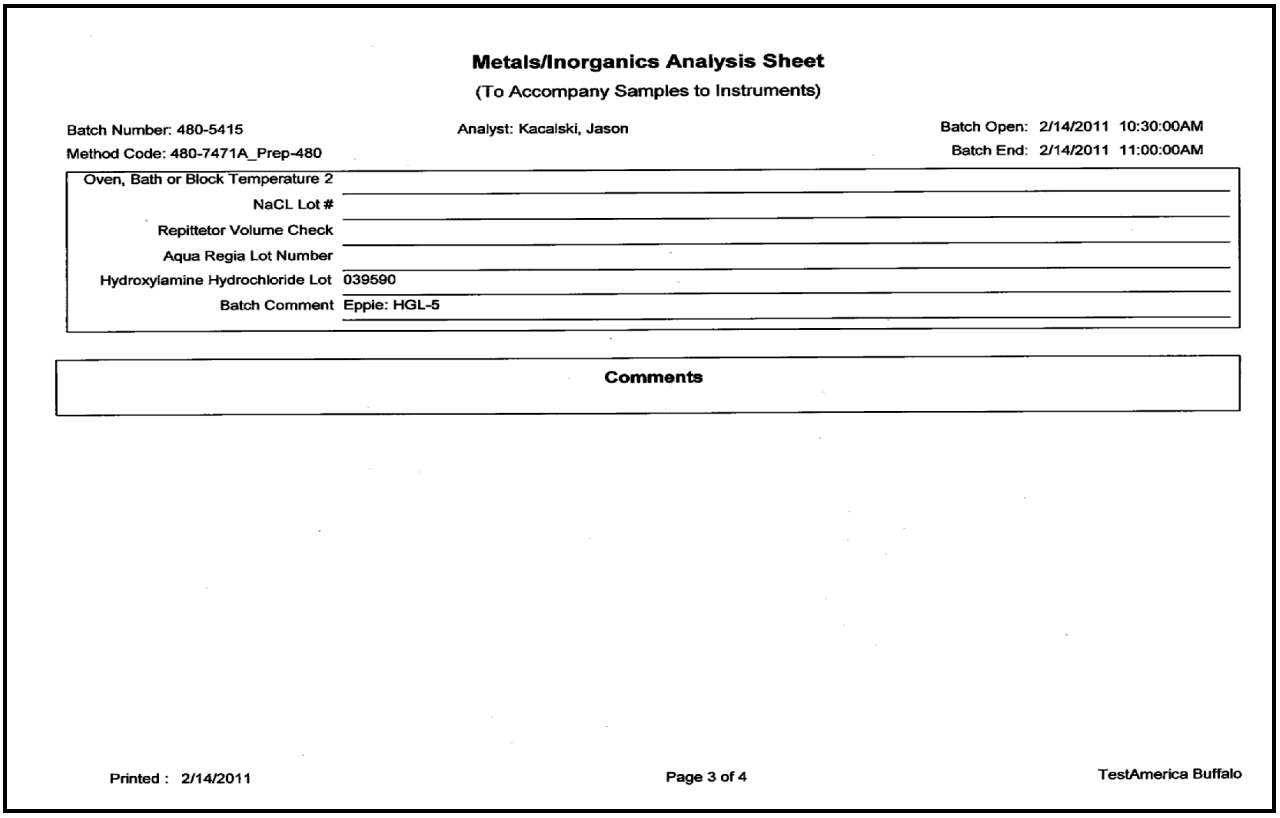
TestAmerica Buffalo

Attachment 3

Sample Soil Digestion Batch Bench Sheet (Page 3 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

Oven, Bath or Block Temperature 2	
NaCL Lot #	
Repittetor Volume Check	
Aqua Regia Lot Number	
Hydroxylamine Hydrochloride Lot	039590
Batch Comment	Eppie: HGL-5

Comments

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Attachment 3

Sample Soil Digestion Batch Bench Sheet (Page 4 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

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCSSRM 480-5415/2	MED_SRM_D066_00001	0.0677 g	50 mL		
480-1553-C-1 MS	MEH_HG2_WKG_00002	2.0 mL	50 mL		
480-1553-C-1 MSD	MEH_HG2_WKG_00002	2.0 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

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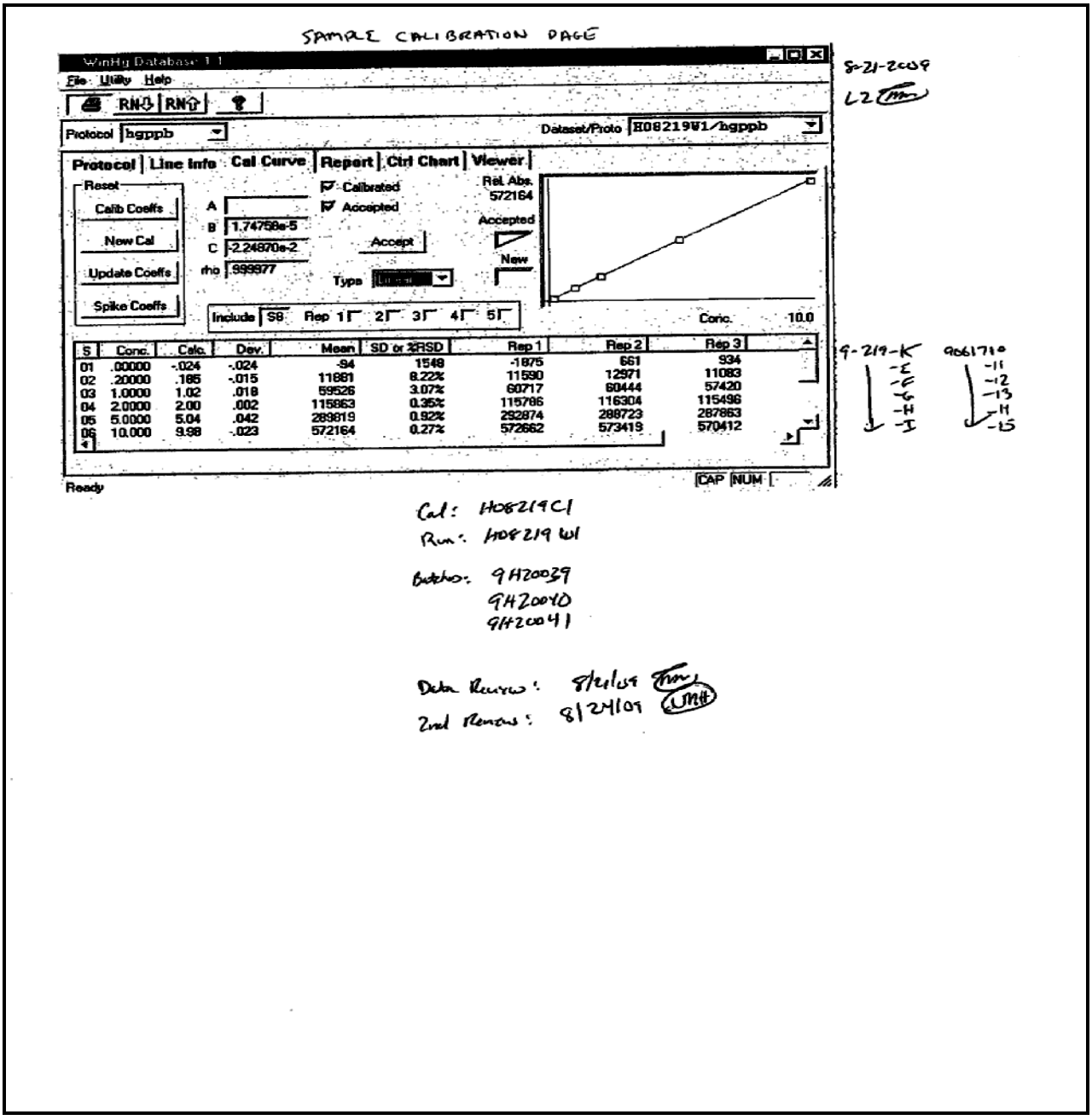
Attachment 4

Example Instrument Calibration Page



Title: Analysis of PCBs
SW846 8082 / 40CFR 608

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

Gary Rudz — 3/27/12	Kenneth E. Kasperek — 3/27/12
Gary Rudz — Date	Kenneth E. Kasperek — Date
Department Manager	Technical Director
Paula Benham — 3/29/12	Christopher Spencer — 3/27/12
Paula Benham — Date	Christopher Spencer — Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This method is used to quantify polychlorinated biphenyls (PCBs) as Aroclors 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, wipe, 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:

0.5 ug/L for water samples	1 Liter	FV=10 ml	8082
0.06 ug/L for low level water samples	1 Liter	FV= 2 ml	608
1 ppM for routine soil samples	2 grams	FV=10 ml	8082
16.7 ug/Kg for soil samples (100% Dry)	30 grams	FV=10 ml	8082
2.5 mg/Kg for oil/waste samples	0.2 grams	FV=10 ml	8082
1 ug/wipe for wipe sample	1 Wipe	FV=40 ml	8082

2.0 Summary of Method

2.1. Wastewater samples: approximately 1 liter of sample is extracted with Methylene chloride using a separatory funnel (SOP ASP-3510B-80) or a continuous liquid-liquid extractor (SOP No. ASP-3520B-85). Soil samples are extracted using approximately 2g of soil/solid sample using sonication (SOP No. ASP-3550B-90) 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.

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 insure 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 chorines 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 (ASP-BF-OP-010 is part of the extraction procedure for all PCB samples.

4.3. Copper cleanup Method 3660 (AGE-BF-GE-005), Gel Permeation Cleanup (ASP-BF-OP-009), Silica Gel Cleanup (ASP-BF-OP-008), and Florisil Cartridge Cleanup (ASP-BF-OP-007) 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. 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.

5.5. 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.

5.6. Primary Materials Used

5.6.1. 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 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
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 degreases 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 Material Safety Data Sheets (MSDS) 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
ZB-5 30m 0.53mm 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

6.1. Instrumentation *

Hewlett Packard 5890 gas chromatograph w/dual ECD detectors
Hewlett Packard 6890 gas chromatograph w/dual uECD detectors or Equivalent
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 (PTFE)

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±2°C in Teflon-sealed amber containers in the dark, unless the manufacturer's storage recommendations differ. These cases will give rise to alternate storage conditions based upon need and availability.

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. Acetone (pesticide grade)

7.6. 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 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.

8.3. Aqueous Samples (8082) 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.

8.4. Aqueous Samples (608) are to be collected in a 1-liter amber glass jar and stored at 4±2°C. Organic preparation is to be performed within 1 year of collection.

8.5. Soil (solid) samples are to be collected in a 4 oz jar and stored at 4±2°C. Organic preparation is to be performed within 14 days of collection.

8.6. Wipe and Oil samples are to be collected in a 4 oz jar and stored at 4±2°C. Organic preparation is to be performed within 28 days of collection.

8.7. Analysis of all extracts is to be performed within 40 days of preparation.

8.8. Holding times specified in project specific quality assurance plans may supersede the above listed method criteria.

8.9. Extracts are stored in an Incubator under refrigeration at 4±2°C.

8.10. For CLP, AFCEE, and USACE samples, the extracts are maintained in secure storage incubator SC# 4

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. For USACE, the method blank must contain concentrations < ½ the reporting limit.

9.1.3. 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 LCS 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). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry.

9.1.4. Limited sample volume can allow for the analysis of a matrix spike blank duplicate instead of a MS and MSD pair.

9.1.5. A laboratory control sample (LCS) must be prepared and analyzed with each batch. A laboratory control sample duplicate (LCSD)) should be prepared if insufficient volume exist for a sample spike duplicate. Spike recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data.

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.

9.2.1.2. 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.

9.2.1.3. 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.

9.2.1.4. 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: 0.005ng, 0.010ng, 0.020ng, 0.030ng, 0.040ng, and 0.05ng.

9.2.1.5. 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).

Calibration Standard Conc.	**RL 8082 Water** (1L fv=10 ml)	**RL 608 Water** (1L fv=2 ml)	**RL 8082 Soil** (30g/100% dry)	**RL 8082 Wipes** (FV=40.0ml)	**RL 8082 Oils** (0.1g fv=10ml)
0.025 ng/ul	**	**0.06 ug/L** *	**	**1.0 ug/Wipe**	**2.5 mg/Kg**
0.05 ng/uL	**0.50 ug/L**	0.10 ug/L	**16.67 ug/kg**	2.0 ug/Wipe	5.0 mg/Kg
0.10 ng/uL	1.0 ug/L	0.20 ug/L	33.3. ug/kg	4.0 ug/Wipe	10 mg/Kg
0.25 ng/uL	2.5 ug/L	0.5 ug/L	83.3 ug/kg	10.0 ug/Wipe	25 mg/Kg
0.50 ng/uL	5.0 ug/L	1.0 ug/L	166 ug/kg	20.0 ug/Wipe	50 mg/Kg
1.0 ng/uL	10.0 ug/L	2.0 ug/L	333 ug/kg	40.0 ug/Wipe	100 mg/Kg
2.0 ng/uL	20.0 ug/L	4.0 ug/L	883 ug/kg	80.0 ug/Wipe	250 mg/Kg

*- Adjusted reflecting MDLV data
** - Not Supported with MDL Studies

9.2.2. Continuing Calibration Verification (CCV)

9.2.2.1. A Continuing Calibration Verifications (CCV) must bracket every 10 samples. Aroclor1660 is used to represent all Aroclors for this purpose.

9.2.3. Instrument Blank (IBLK)

9.2.3.1. An Instrument Blank (IBLK) is analyzed immediately after the Continuing Calibration Verification (CCV) to verify the absence of any potential carry-over or contamination.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows

9.2.4.1.1. The Initial RTWindow 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.

9.2.4.1.2. 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.

9.2.4.1.3. The Chrom 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.

9.2.4.2. Calibration Model

9.2.4.2.1. 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

9.2.4.2.2. 1st Order Calibrations do not prove linearity through zero, and therefore does not allow for the use of single point standards to be used to quantify any other Aroclors of interest.

9.2.4.2.3. 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.

9.2.4.2.4. If RSD $\leq$20% 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.

9.2.4.2.5. 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%.

9.2.4.2.6. For each Aroclor, three to five major chromatographic peaks are chosen that represent the key to the patterns present in each particular Aroclor for quantification. Four – 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.

9.2.4.2.7. ***Is has been found that "Unique" Peaks for each Aroclor do not always exist, the fact is that many mixes containing all the congeners for another Aroclor.***

9.2.4.2.8. Due to this, the following combinations of Aroclors cannot be identified simultaneously in a given sample:

9.2.4.2.9. Aroclors: 1016, 1232, & 1242 will not have unique peaks that meet all criteria when compared to one another.

9.2.4.2.10. Aroclors: 1221 & 1232 will not have unique peaks that meet all criteria when compared to one another.

9.2.4.2.11. Aroclors: 1242 & 1248 will not have unique peaks that meet all criteria when compared to one another.

9.2.4.2.12. Aroclors: 1260 & 1262 will not have unique peaks that meet all criteria when compared to one another.

9.2.4.2.13. 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.

9.2.4.2.14. 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 try 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.

9.2.4.2.15. The Percent Relative Standard Deviation for each surrogate must be $\leq$20%, or have a Correlation Coefficient "R" $\geq$0.995 (R squared $\geq$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.

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. An acceptable ICC curve is run for Ar1660 and surrogates, and single point calibrations are then run for all remaining Aroclors. 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/CCV 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 Aroclor standard.

10.1.4.1. The response factor of the ICV must be ±15% D for Aroclors 1016 & 1260. The ICVs and CCVs may >15% biased high to confirm non-detects. ICVs and CCVs that are biased >15% low will require a sample and system evaluation to determine if the effects are temporary or lasting.

10.1.4.2. In the temporary case, the instrument should be baked out and allowed to come to equilibrium to check compliance.

10.1.4.3. 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.

10.1.4.4. 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.

10.1.4.5. Sample analysis which continually produces reduced response upon re-analysis can be dealt with in several ways

10.1.4.5.1. The extracts should be check for possible further cleanup and/or dilution to minimize these effects.

10.1.4.5.2. 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.

10.1.4.5.3. 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.

10.1.4.6. 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:

10.1.4.6.1. Dilutions due to these facts, not based upon matrix, positives, color, or any other visual data.

10.1.4.6.2. Review of Report Level requirements and submittal of data as analyzed, with comment that the data is to be considered bias low

10.1.4.6.3. 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 as in 9.2.4.1.1 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 CCV midpoint standard to confirm full pattern recognition when needed. These additional standards are not subject to quantifiable verification.

10.1.6.1. The total response factor for each Aroclor in the CCV must be less than or equal to ±15% D. (Individual peaks may be 15% > 25%, as long as the total Aroclor amount or average is </= 15%.) All data is acceptable as long as it is bracketed by an acceptable ICV and CCV, or CCV and CCV.

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 3510A (waters SOP # BF-OP-003) and 3550B (soils SOP # BF-OP-005).

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. For USACE, the method blank must not contain compounds at levels > ½ the report level.

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: (lab control sample) 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/LCSD/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry. 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.

10.3.4. MS: A matrix spike sample must be set for one in every batch of 20 samples if sufficient sample volume exists. 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 LCS.. 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.

10.3.5. MSD: Along with every matrix spike sample, a duplicate MS must also be set if volume exists. This sample is the matrix spike duplicate (MSD). Minimum acceptance criteria are <50% 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 LCS.

10.3.6. 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

10.4. Example Analysis Queue

Instrument Counter	Number of Runs	ID	Comments
1-2	*2*	*Priming Runs*	*Weekly Startup*
3-12	*9*	*All Individual Aroclors*	*CCV(66), RT & Pattern Check*
13	*1*	*ICM3*	*Instrument Blank*
14-23	*10*	*Samples*	
24-25	*2*	*ICM66 & ICM3*	*CCV & Inst Blank – Every 10 Injections*
26-35	*10*	*Samples*	
36-37	*2*	*ICM66 & ICM3*	*CCV & Inst Blank – Every 10 Injections*
1-2	*2*	*Priming Runs*	*Daily Startup*
3-4	*2*	*ICM66 & ICM3*	*CCV & Inst Blank*
5-14	*10*	*Samples*	
15-16	*2*	*ICM66 & ICM3*	*CCV & Inst Blank – Every 10 Injections*
17-26	*10*	*Samples*	
27-28	*2*	*ICM66 & ICM3*	*CCV & Inst Blank – Every 10 Injections*

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)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

Concentration = 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 TALs before 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

$$\frac{\text{Peak(n)ng + peak(n+1)ng + peak(n+2)ng +}}{\text{Total Peaks}} = \text{ng amount of sample}$$

Converting ng amount to ug/Kg, ug/L and ug/wipe

$$\text{ug/Kg} = \frac{(\text{ng}) \times (\text{final volume in ml}) \times (\text{dilution factor})}{1000} \times \frac{}{(\text{injection vol. in ul}) \times (\text{sample wt.}) \times (\text{\% Dry})}$$

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

12.1.1. Method Detection Limit Study (MDL)

12.1.1.1. MDL studies are performed annually on a matrix and instrument type basis

12.1.1.2. 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.

12.1.1.3. MDL studies are to be preformed using Aroclor1016/1260 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.

12.1.1.4. 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.

12.1.2. Demonstration of Capabilities

12.1.2.1. 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/LCS.

12.1.2.2. 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

12.1.2.3. A continuing demonstration of capability (DOC) will be performed on an annual basis for each analyst, for each operational method they run and analyze.

12.1.3. Training Requirements

12.1.3.1. 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. Method 8082 U.S. Environmental Protection Agency, Office of Solid Waste and Energy Response, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd edition, SW-486, update III, Dec. 1996.

16.0 Method Modifications: N/A

17.0 Attachments

Attachment A: Job Summary Sheet and Data Review Checklist

18.0 Revision History

- Revision 2, dated March 29, 2012
  - Changed QA Manager, signature added.
  - Updated all LIMs references from Element to TALs.
  - Replaced all MSB references with LCS.
  - Section 9.2.4.2 updated calibration model to reflect Average Cal Factor needed to use single point ICALs for Aroclors.
  - Clarified ICV and CCV references pertaining to TALs.

- Revision 1, dated March 18, 2010
  - Replaced WO summary example with current document.
  - Removed all LIMs references from Aims to Element.

- Revision 0, dated March 31, 2008
  - Integration for TestAmerica and STL operations.

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 7/30/13 Denise Giglia Date Department Manager	Kenneth Kasperek 7/30/13 Kenneth Kasperek Date EHS Manager
7/30/13 Brad Prinzi Date Quality Assurance Manager	7/30/13 Christopher Spencer Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods 8260C -5 mL aqueous purge, 8260C - 25mL 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 or 25mL purge (concentrations adjusted accordingly).

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 20 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
  - Carbpack B
  - Carboxen 1000
  - Carboxen 1001
- OI #10
  - Tenax
  - Silica Gel
  - 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 3.0μm

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 three 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 54 Component Mix (See Table 9 for component list) is purchased at a concentration of 2000ug/ml.

7.3.1.3 8260+ Mix (See Table 10 for component list) is purchased and is composed of four separate mixtures.

- 8260+ Mix #1 is purchased at a concentration of 1000ug/ml.
- 8260+ Mix #2 is purchased at a concentration of 5000ug/ml.

- 8260+ Mix #3 is purchased at a concentration of 20000ug/ml.
- 8260+ Mix #4 is purchased at a concentration of 5000ug/ml.

7.3.2 **Stock Calibration Verification Mix** – Is composed of two different mixtures.

7.3.2.1 The Second Source Mix (See Table 11 for component list) is purchased at a concentration of 2000ug/ml.

7.3.2.2 The 8260+ Second Source Mix (See Table 12 for component list) is purchased and is composed of two separate mixtures.

- 8260+ Second Source Mix #1 is purchased at a concentration of 1000ug/ml.
- 8260+ Second Source Mix #2 is purchased at a concentration of 5000ug/ml.

7.3.3 **Stock Internal Standard Solution** – A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and 1,4-Difluorobenzene in Methanol is purchased at a concentration of 2500ug/ml.

7.3.4 **Stock System Monitoring Solution** – A mixture of Toluene-D8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4 in Methanol is purchased at a concentration of 2500ug/ml.

7.3.5 **Stock Matrix Spike Solution** – A 17 component mixture of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, benzene, 1,2,4-trimethylbenzene, 1,1-dichlorethane, 1,2-dichlorobenzene, 1,2-dichloroethane, cis-1,2-dichloroethene, ethylbenzene, m-xylene, o-xylene, p-xylene, methyl tert butyl ether, tetrachloroethene, and trans-1,2-dichloroethene in Methanol is purchased at a concentration of 2500ug/ml.

7.3.6 **Stock BFB Solution** – A solution of 4-Bromofluorobenzene in Methanol is at a concentration of 25000ug/ml.

7.4 **Secondary IS and System Monitoring Calibration Dilution Standards** – these solutions are used for the manual injections required to prepare the initial calibration.

7.4.1 **Internal Standard Solution** - 80ul of stock standard IS solution (2500ug/ml) 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 100ng/ul.

7.4.2 **System Monitoring Compound Solution** - 80ul of stock standard Surrogate solution (2500ug/ml) is added to approximately 1 ml of purge and trap methanol in a 2 ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol for a final concentration of 100ng/ml.

7.4.3 To calculate appropriate expiration dates, refer to “Standards Traceability and

Preparation Logbooks".

7.5 Working Standards

7.5.1 Intermediate Calibration Solution (Three individual mixtures)

7.5.1.1 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.

7.5.1.2 500ul of stock standard 54 Component Mix solution (2000ug/ml) is added to approximately 9ml 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 for a final concentration of 100ng/ul.

7.5.1.3 1000ul of each of the four stock standard 8260+ 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.

7.5.2 **Matrix Spike Solution** - 100ul of stock standard 17 component solution (2500ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5 ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

7.5.3 **A Full List Matrix Spike Standard** is made from stock Calibration Verification Standards and is composed of two mixes.

7.5.3.1 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.

7.5.3.2 200ul of each of the two stock standard 8260+ Second Source Mixes are added to approximately 1ml of purge and trap methanol in a 2ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol.

7.5.4 **Working Internal Standard and System Monitoring Compound Solutions** – for auto injection by instrument.

7.5.4.1 **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.

7.5.4.2 **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.

7.5.5 **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.

7.5.6 **Working Initial Calibration Standards**

7.5.6.1 **Water: 25 mL**

7.5.6.1.1 20ul, 10ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 15ul, 5ul and 0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of three 50ml volumetric flasks for multi-point surrogate calibration. The flasks are brought to volume with reagent water to prepare the 40, 20 and 10 ug/L standards respectively. For single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.5.6.1.2 4ul and 1ul each of Intermediate Calibration Solution (7.5.1) and System Monitoring Compound Solution (7.4.2) are added to reagent water in 100 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 4ug/L and 1ug/L standards respectively.

7.5.6.1.3 Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.2 **Water: 5 mL (5 point curve)**

7.5.6.2.1 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) 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. For single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.5.6.2.2 1ul of each Intermediate Calibration Solution (7.5.1) and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to

reagent water in a 100ml volumetric flask. The flask is brought to volume with reagent water to prepare the 1ug/L standard.

7.5.6.2.3 The standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.3 Water: 5 mL (6 point curve)

7.5.6.3.1 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) 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. For single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.5.6.3.2 5ul and 1ul of each Intermediate Calibration Solution (7.5.1) and 5.0ul and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of two 100ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 5.0 and 1.0ug/L standards respectively.

7.5.6.3.3 Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.4 Soil: (5 point curve)

7.5.6.4.1 100ul, 50ul, 25ul, 10ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of five 50ml volumetric flasks for multi-point surrogate calibration. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20 and 5 ug/kg, standards respectively. For single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.5.6.4.2 5 ml of each standard is then transferred into five individual 40ml vials and loaded onto the auto sampler.

7.5.6.5 Soil: (6 point curve)

7.5.6.5.1 100ul, 50ul, 25ul, 10ul, 5ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul, 5ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of six 50ml volumetric flasks for multi-point surrogate calibration. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20, 10 and 5

ug/kg, standards respectively. For single point surrogate calibration, the instruments' auto sampler introduces 25ug/L of System Monitoring Compounds into all calibration points.

7.5.6.5.2 5 ml of each standard is then transferred into six individual 40ml vials and loaded onto the auto sampler.

7.5.7 Continuing Calibration Standard

7.5.7.1 Water: 25 ml

7.5.7.1.1 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 10ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.5.7.2 Water: 5 ml

7.5.7.2.1 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.

7.5.7.3 Soil:

7.5.7.3.1 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.

7.6 Storage of Standards

7.6.1 Stock standards are stored in flame sealed ampoules at 22^0 C to -20^0 C according to the vendor's specifications.

7.6.2 Secondary dilution standards are stored in Teflon-sealed crimp cap vials at $< 0^0$ C.

7.6.3 Aqueous standards are stored in Teflon-sealed vials at 4^0 C $\pm$ 2^o 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±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±2^{0}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 with 8ul of 50ng/ul or 4ul of 100ng/ul matrix spike standard for 25ml purge and 22ul of 50ng/ul or 11ul of 100ng/ul for the 5ml purge. This corresponds to a final concentration in the samples of 10ug/L and 25ug/L respectively. Analysis proceeds according to procedures described for water analysis.

8.5.2 Low Level Soil/Sediment Samples: 5ul of 50ng/ul or 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 10ug/L for 25ml analysis, 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 10ug/L for 25ml analysis, 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 Routine compounds included in the LCS are:

1,1-Dichloroethene; Chlorobenzene; Toluene; Benzene; Trichloroethene; 1,2,4-Trimethylbenzene; 1,2-Dichlorobenzene; 1,2-Dichloroethane; 1,1-Dichloroethane; cis-1,2-Dichloroethene; Ethylbenzene; m-Xylene; p-Xylene; o-Xylene; t-Butyl methyl ether; Tetrachloroethene; trans-1,2-Dichloroethene

9.2.6 When required, 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.

9.2.7 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.

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 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.

9.4.1.4.1 Following the analysis of an acceptable initial calibration curve, an aliquot of this independent standard is analyzed at the CCV level.

9.4.1.4.2 Recoveries of all compounds shall fall within ±30% of the expected values.

9.4.2 Technical Acceptance Criteria For Continuing Calibration

9.4.2.1 **Minimum Response Factors** - A 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 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 criteria. If more than 20% of the compounds fail to meet this criteria a new calibration is required.

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. When required, the LCS will be spiked with all compounds of interest, otherwise spiked to include a minimum of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, benzene, 1,2,4-trimethylbenzene, 1,2-dichlorobenzene, 1,2-dichloroethane, 1,1-

dichloroethane, cis-1,2-dichloroethene, ethylbenzene, m-xylene, p-xylene, o-xylene, t-butyl methyl ether, tetrachloroethene and trans-1,2-dichloroethene. 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 Perflurotributylamine (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)
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)
VSTD005	MeOH	100ng/ul	2.5	50	5
VSTD010*	MeOH	100ng/ul	5	50	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

25 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. Water (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD004	MeOH	100ng/ul	4	100	4
VSTD010	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD040	MeOH	100ng/ul	20	50	40

* optional 6th point for the initial calibration

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, 10ug/L for 25ml 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 (10ug/L for 25ml or 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 **25ml Water:** 5ul of target compound mixture is added to a 50ml volumetric flask. A 25ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.

10.2.4.3 **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. 25ml analysis requires a final concentration of 10ug/L. 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 $\pm$ 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 190C to 250^{0}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^{0}C to 260^{0}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^{0}C $\pm$ 1^{0}C then purged for 11 $\pm$ 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 >= -7degrees 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 with in 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 40^0C $\pm$ 1^0C then purged for 11 $\pm$ 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 is shaken for 2 minutes.

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 >= -7degrees 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 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:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

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:

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

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.

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

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)
Cis = Concentration of the internal standard
Cx = 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:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n}(\chi i - \bar{\chi})2}{n-1}}$$

Where,
Xi = each individual value used to calculate the mean
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:

$$\%\text{Difference} = \frac{RRFc - RRFi}{RRFi} \times 100$$

Where,
RRFc = Relative response factor from continuing calibration standard
RRFi = 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^{0}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.

$$\%\text{moisture} = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

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 form 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:

$$\frac{\text{ul most conc. extract used to make dilution + ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

(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).

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, 4th 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 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

- Initial Version

Table 1: Compounds Determined by Method 8260C

Compound	CAS No.[b]	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
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:

Bromombenzene
n-Butylbenzene
sec-Butlybenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
cis-1,2-Dichloroethene
Di-isopropyl ether
tert-amyl methyl ether
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
tert-butyl ethyl ether

Table 2. BFB Key Ions and Ion Abundance Criteria

mz	Required Intensity (relative abundance)
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 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
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

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
trans-1,4-Dichloro-2-butene	53	88,75
Dichlorodifluoromethane	85	87
1,1-Dichlorothane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichlorothene	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
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

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
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
1,4-Difluorobenzene	114	
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

1,1-Dimethoxyethane*	Bromomethane
1,2-Dibromo-3-chloropropane (DBCP)	Carbon Disulfide
1,4-Dioxane*	Chloroethane
2-Butanone (MEK)	Cyclohexanone*
2-Chloroethylvinyl ether	Dichlorodifluoromethane
2-Nitropropane*	Iodomethane
4-Methyl-2-pentanone (MIBK)	Methyl Acetate
Acetone	Propylene Oxide*
Acrolein	trans-1,4-Dichloro-2-butene

***** Indicates “Add” compounds that are not routinely spiked for in LCS/MS/SD

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

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

GCMS VOA Data Review Checklist
Rev. 1 October 24, 2012

GCMS VOA Work Order Summary

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

Certificate of Composition

DESCRIPTION: Volatile Organic Compounds Mix 6

CATALOG NO.: 48799-U

MFG DATE: Nov-2005

LOT NO.: LB34727

EXPIRATION DATE: Feb-2007

MVSC 72 8-20

MVSC 73 1-7

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
BROMOMETHANE	74-83-9	99.9 (a)	2000	LB22203
CHLOROETHANE	75-00-3	98.7 (a)	2000	LB29285
CHLOROMETHANE	74-87-3	99.9 (a)	2000	LA66620
DICHLORODIFLUOROMETHANE	75-71-8	99.9 (a)	2000	LB24923
TRICHLOROFLUOROMETHANE	75-69-4	99.9 (a)	2000	LA79530
VINYL CHLORIDE	75-01-4	99.9	2000	LB18727

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

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Table 9. 54 Component Mixture

Certificate of Analysis

MVSC 19 15-20
20 1-4

PAGE 1 OF 2

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
BENZENE	71-43-2	99.9	2000	2000	+/-	15.1	LB03979
BROMOBENZENE	108-86-1	99.9	2000	2009	+/-	17.4	LA97903
BROMOCHLOROMETHANE	74-97-5	99.7	2000	1967	+/-	33.3	LA67395
BROMODICHLOROMETHANE	75-27-4	99.9	2000	2103	+/-	0.1	LB15447
BROMOFORM	75-25-2	99.9	2000	1974	+/-	38.7	LB15898
CARBON TETRACHLORIDE	56-23-5	99.9	2000	1960	+/-	32.4	LA55581
CHLOROBENZENE	108-90-7	99.9	2001	2029	+/-	14.3	LB09884
CHLOROFORM	67-66-3	99.9	2000	2000	+/-	18.8	LA55585
CIS 1,3-DICHLOROPROPENE (Z)	10061-01-5	96.1	2000	2036	+/-	12.1	LA60646
CIS-1,2-DICHLOROETHYLENE	156-59-2	97.6	2000	1947	+/-	26.7	LA97197
DIBROMOCHLOROMETHANE	124-48-1	99.9	2001	2022	+/-	11.2	LA87237
DIBROMOMETHANE	74-95-3	99.8	2000	2000	+/-	33.6	LA39031
ETHYLBENZENE	100-41-4	99.5	2000	2040	+/-	8.0	LA40866
HEXACHLOROBUTADIENE	87-68-3	98.2	2001	1946	+/-	45.0	LA95300
ISOPROPYLBENZENE (CUMENE)	98-82-8	99.0	2000	2012	+/-	17.3	LB01119
M-XYLENE (5)	108-38-3	99.8	2001	*****			LB15074
METHYLENE CHLORIDE	75-09-2	99.9	2000	1957	+/-	28.9	LA88418
N-BUTYLBENZENE	104-51-8	98.7	2000	1996	+/-	25.3	LB09309
N-PROPYLBENZENE	103-65-1	99.9	2001	2028	+/-	15.6	LA92696
NAPHTHALENE	91-20-3	99.9	2000	1950	+/-	39.5	LA97766
O-XYLENE	95-47-6	99.5	2000	2022	+/-	9.8	LB08117
P-ISOPROPYLTOLUENE	99-87-6	99.9	2000	1986	+/-	20.7	LA41611
P-XYLENE (5)	106-42-3	99.9	2000	*****			LB04801
SEC-BUTYLBENZENE	135-98-8	99.4	2000	1993	+/-	31.6	LA51283
STYRENE	100-42-5	99.9	2001	2012	+/-	11.8	LB09037
TERT-BUTYLBENZENE	98-06-6	99.9	2000	1981	+/-	21.8	LB09550
TETRACHLOROETHENE	127-18-4	99.9	2001	2029	+/-	29.4	LB05248

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

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SUMMA

Certificate of Analysis

MUSCM 15-20
20 1-4

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
TOLUENE	108-88-3	99.7	2001	2020	+/-	15.8	LA90411
TRANS 1,3-DICHLOROPROPENE (E)	10061-02-6	98.5	2000	2052	+/-	12.9	LB06449
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.9	2000	1910	+/-	36.2	LB02428
TRICHLOROETHYLENE	79-01-6	98.5	2001	1980	+/-	20.2	LB04303
1,1-DICHLOROETHANE	75-34-3	97.0	2000	1968	+/-	32.1	LA54711
1,1-DICHLOROETHYLENE	75-35-4	99.9	2000	1980	+/-	46.1	LB04593
1,1-DICHLOROPROPENE	563-58-6	98.0	2000	1958	+/-	20.8	LB12558
1,1,1-TRICHLOROETHANE	71-55-6	99.9	2000	1973	+/-	26.8	LB14220
1,1,1,2-TETRACHLOROETHANE	630-20-6	99.1	2001	2000	+/-	16.1	LB01555
1,1,2-TRICHLOROETHANE	79-00-5	99.3	2000	2038	+/-	12.6	LB03464
1,1,2,2-TETRACHLOROETHANE	79-34-5	97.5	2000	1974	+/-	31.7	LA86969
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	97.9	2000	1978	+/-	43.5	LB06608
1,2-DIBROMOETHANE	106-93-4	99.6	2001	2029	+/-	0.1	LA87068
1,2-DICHLOROBENZENE	95-50-1	99.9	2000	2008	+/-	29.2	LA96474
1,2-DICHLOROETHANE	107-06-2	99.9	2000	1974	+/-	25.7	LA88777
1,2-DICHLOROPROPANE	78-87-5	99.9	2000	2019	+/-	9.6	LB08115
1,2,3-TRICHLOROBENZENE	87-61-6	99.75	2000	1962	+/-	18.9	LA50762
1,2,3-TRICHLOROPROPANE	96-18-4	99.1	2000	2006	+/-	17.8	LA39379
1,2,4-TRICHLOROBENZENE	120-82-1	98.6	2000	1957	+/-	52.1	LB12944
1,2,4-TRIMETHYLBENZENE	95-63-6	98.2	2000	2000	+/-	22.0	LA39081
1,3-DICHLOROBENZENE,	541-73-1	99.9	2001	2013	+/-	16.7	LA72024
1,3-DICHLOROPROPANE	142-28-9	99.9	2000	2024	+/-	11.8	LB00875
1,3,5-TRIMETHYLBENZENE	108-67-8	99.0	2000	2011	+/-	13.6	LA94493
1,4-DICHLOROBENZENE	106-46-7	99.9	2000	1992	+/-	16.2	LA50188
2-CHLOROTOLUENE	95-49-8	99.9	2000	2005	+/-	23.6	LA95842
2,2-DICHLOROPROPANE	594-20-7	98.3	2000	1968	+/-	19.4	LB01750
4-CHLOROTOLUENE	106-43-4	99.9	2001	1990	+/-	15.0	LB05252

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

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Table 10. 8260 + Mix

8260X #1A MUSL 5 1/720

Chemical Standard Batch Sheet
Lot #: A042263

Catalog #: 552504A	**Target:** 1000 - 40000 ug/ml	
Description: Custom Volatiles Standard Mix A		
Solvent: P&T Methanol	**Solvent Lot:** 44337	**Final Volume:** 100 ml

Made by: Joe Tallon	**Date:** 1/4/2006 8:09:50A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:49:12/	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Carbon disulfide	75-15-0	FA1A5D	J11J02	0.99	1,000.00	100.00	100.00	1,000.00
Methyl-tert-butyl ether (	1634-04-4	FA1B6C	10660BD	0.97	1,000.00	100.00	100.00	1,000.00
Iodomethane (methyl	74-88-4	FA1C2A	13906AB	0.99	1,000.00	100.00	100.00	1,000.00
Ethyl methacrylate	97-63-2	FA1C1D	09316HC	0.99	1,000.00	100.00	100.00	1,000.00
Tetrahydrofuran	109-99-9	FA1B8B	01057MC	0.99	5,000.00	500.00	500.00	5,000.00
trans-1,4-dichloro-2-butene	110-57-6	FA1C1C	160-22DD	0.99	5,000.00	500.00	500.00	5,000.00
Acetonitrile	75-05-8	FA1B13A	12067KC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	1,000.00	100.00	100.00	1,000.00
Methyl acetate	79-20-9	FA1C11C	47640/1	0.99	1,000.00	100.00	100.00	1,000.00
Methylcyclohexane	108-87-2	FA1E4A	02759BC	0.99	1,000.00	100.00	100.00	1,000.00
Cyclohexane	110-82-7	FA1C7A	03145KB	0.99	1,000.00	100.00	100.00	1,000.00

8260+ #1B MVSC5 1-720

Chemical Standard Batch Sheet
Lot #: A042264

Catalog #: 552504B	**Target:** 5000 ug/ml	
Description: Custom Volatiles Standard Mix B		
Solvent: P&T Methanol	**Solvent Lot:** A041266	**Final Volume:** 50 ml

Made by: Joe Tallon	**Date:** 1/4/2006 8:30:59A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:54:16A	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
2-Chloroethyl vinyl ether	110-75-8	FA1A11D	03206CI	0.99	5,000.00	250.00	250.00	5,000.00

Certificate of Composition

5260 + #3

MVSC 42 4 → 13

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20460869 LOT NO.: LB25705 MFG DATE: Dec-2004

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20008 +/- 100.0	LB21530
ACRYLONITRILE	107-13-1	99.9	20000 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

SUPELCO
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8260 #4 MVSC 5 1→10

Chemical Standard Batch Sheet
Lot #: A042268

Catalog #: 556843	**Target:** 5000 ug/ml	
Description: Custom Vinyl Acetate Standard		
Solvent: P&T Methanol	**Solvent Lot:** A038421	**Final Volume:** 25 ml

Made by: Joe Tallon	**Date:** 1/4/2006 9:40:21A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:58:29A	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Vinyl acetate	108-05-4	FA1A9A	08831CW	0.99	5,000.00	125.00	125.00	5,000.00

MVSC 23 6→20
24 1→75

Gravimetric Certificate

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 552501 Lot No.: A044128
Description: Custom Ketones Standard
Expiration Date[1]: March 2008 Storage: Freezer

Component #	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	2-Butanone (MEK)	78-93-3	99%	5,000.00 ug/ml	+/-0.08 %
2	2-Hexanone	591-78-6	99%	5,000.00 ug/ml	+/-0.08 %
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5,000.00 ug/ml	+/-0.08 %
4	Acetone	67-64-1	99%	5,000.00 ug/ml	+/-0.08 %

Solvent: P/T Methanol/Water (90:10)

F. Joseph Jaflon - Mix Technician Balance: 1119141429

[1] ...tion date of the unopened ampul stored at recommended temperature.
[2] ... was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the ...earest 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 preperation with balance calibration verified using NISTraceable weights (seven mass levels).
[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured under Restek's ISO 9001 Registered Quality System Certificate #FM80397

Table 11. Second Source 60 Component Mixture

ULTRA SCIENTIFIC
Analytical Solutions

60 Comp S. Source

Certificate of Analysis MUSC 71

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 1 of 3

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	CAS#	Analyte Lot	True Value
bromochloromethane	000074-97-5	JS-16015HS	2006 ± 10 µg/mL
bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 µg/mL
bromoform	000075-25-2	DU-06126KS	2006 ± 10 µg/mL
carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
chloroform	000067-66-3	BS-03041BS	2006 ± 10 µg/mL
dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
dibromomethane	000074-95-3	EM-01514TJ	2006 ± 10 µg/mL
methylene chloride	000075-09-2	44267	2006 ± 10 µg/mL
trichlorofluoromethane	000075-69-4	DR-16417BR	2006 ± 10 µg/mL
1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethane	000075-34-3	64552/1	2006 ± 10 µg/mL
1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
1,1-dichloroethene	000075-35-4	01218EC	2007 ± 10 µg/mL
cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
1,1,1,2-tetrachloroethane	000630-20-6	CO-12312LI	2006 ± 10 µg/mL
1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
1,1,1-trichloroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL
1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene	000079-01-6	KN-08846KN	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2005 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2005 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2006 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34251-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	2005 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	12020TF	2006 ± 10 µg/mL
naphthalene	000091-20-3	14205KB	2005 ± 10 µg/mL
benzene	000071-43-2	31072	2006 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2005 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2006 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2005 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2006 ± 10 µg/mL
styrene	000100-42-5	MQ-11229MQ	2005 ± 10 µg/mL
toluene	000108-88-3	43045	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2007 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CJ	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
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ACCREDITED
ISO 17025
Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2005 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2005 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2006 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2009 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2009 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

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www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

Table12. Second Source 8260 + Mixture

8260+#1 Sec Source MVSC6 1→10

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687608 LOT NO.: LB35787 EXPIRATION DATE: Jan-2007

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONITRILE	75-05-8	99.9	40001 +/- 200.0	LB34175
CARBON DISULFIDE	75-15-0	99.9 (a)	999 +/- 5.0	LB09107
CYCLOHEXANE	110-82-7	99.9	1000 +/- 5.0	LB18076
ETHYL METHACRYLATE	97-63-2	99.3	1002 +/- 5.0	LA29651
FREON 113	76-13-1	99.9 (b)	1001 +/- 5.0	LA33286
METHYL ACETATE	79-20-9	98.1	1001 +/- 5.0	LB32233
METHYL CYCLOHEXANE	108-87-2	99.8	1001 +/- 5.0	LB06982
METHYL TERT-BUTYL ETHER	1634-04-4	99.9	1002 +/- 5.0	LB34302
TETRAHYDROFURAN	109-99-9	97.4	4999 +/- 25.0	LA58136
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6	98.2	5002 +/- 25.0	LB10202
1-CHLOROHEXANE	544-10-5	99.9	1000 +/- 5.0	LB18907

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector FPD
b) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

8260+#'s Sec Source MVSC6 11→20

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687609 LOT NO.: LB35788 EXPIRATION DATE: Jan-2007

SOLVENT: DEIONIZED WATER 50 %
METHANOL 50 %

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONE	67-64-1	99.9	5004 +/- 25.0	LB31953
IODOMETHANE	74-88-4	99.9	1004 +/- 5.0	LA73149
VINYL ACETATE	108-05-4	99.9	5002 +/- 25.0	LB31606
2-BUTANONE	78-93-3	99.9	5004 +/- 25.0	LB19842
2-HEXANONE	591-78-6	99.9	5004 +/- 25.0	LB08447
4-METHYL-2-PENTANONE	108-10-1	99.9	5004 +/- 25.0	LA99226

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

Certificate of Analysis

MVSC 66 3-7

DESCRIPTION: 2-Chloroethyl vinyl ether

CATALOG NO.: 40017 MFG DATE: Feb-2005

LOT NO.: LB27794 EXPIRATION DATE: Feb-2008

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3) CONCENTRATION	STD DEV	SUPELCO LOT NO
2-CHLOROETHYL VINYL ETHER	110-75-8	99.9	5000	5000	+/- 55.9	LB01239

(1) Determined by capillary GC-FID, unless otherwise noted.
(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

Certificate of Composition

8260 Mix #3 Sec. Source
MVSC 7
1-7/10

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687606 LOT NO.: LB35789 EXPIRATION DATE: Jul-2006

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)		SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20012	+/- 100.1	LB21530
ACRYLONITRILE	107-13-1	99.9	20008	+/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441

Table 13. 8260 Add Mixture

MVSC 71-18-20
72-01-07

Chemical Standard Batch Sheet
Lot #: A042005

Catalog #: 552546	Target: 2000-80000 ug/ml	
Description: Custom Volatiles Standard		
Solvent: P&T Methanol	Solvent Lot: 44337	Final Volume: 100 ml

Made by: Ryan Miller	Date: 12/19/2005 10:12:4
Tested by:	Date:
	By: Date:
Packaged by: / SLB / JG	Date: 12-20-05 No. Units: 12
Balance Used: AT400	Serial #: 1113372841

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Allyl chloride (	107-05-1	FA1B13D	00305HO	0.99	2,000.00	200.00	200.00	2,000.00
Chloroprene	126-99-8	FA1D8B	051215JLM	0.99	2,000.00	200.00		0.00
Pentachloroethane	76-01-7	FA1C3B	OGL01	0.98	2,000.00	200.00	200.00	2,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	2,000.00	200.00	200.00	2,000.00
Dichlorodifluoromethane	75-71-8	HOOD	A042007	0.99	2,000.00		4.20 (ml)	1,978.41
Dichlorofluoromethane	75-43-4	HOOD	A042008	0.99	2,000.00		3.10 (ml)	1,974.39
Chlorodifluoromethane	75-45-6	VOA Lab	A042009	0.99	2,000.00		2.40 (ml)	2,016.62
Ethyl acetate	141-78-6	FA1C5B	11073ED	0.99	2,000.00	200.00	200.00	2,000.00
Diisopropyl ether (DIPE)	108-20-3	FA1C2B	13450CB	0.99	2,000.00	200.00	200.00	2,000.00
Hexachloroethane	67-72-1	RA1B6D	12719A0	0.99	2,000.00	200.00	200.00	2,000.00
Methyl methacrylate	80-62-6	FA1C2D	09505TO	0.99	2,000.00	200.00	200.00	2,000.00
Methacrylonitrile	126-98-7	FA1C2C	04406MI	0.99	2,000.00	200.00	200.00	2,000.00
Diethyl ether (ethyl ether)	60-29-7	FA1C1A	17676TQ	0.99	2,000.00	200.00	200.00	2,000.00
2-Nitropropane	79-46-9	RA1C11C	04609PN	0.98	10,000.00	1,000.00	1,000.00	10,000.00
Pr nitrile	107-12-0	FA1C3D	10101EB	0.98	20,000.00	2,000.00	2,000.00	20,000.00
Cyclohexanone	108-94-1	RA1D2B	10513PA	0.99	20,000.00	2,000.00	2,000.00	20,000.00
ert-Butanol (TBA)	75-65-0	RA1H2D	06648PC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
l-Butanol	71-36-3	FA1G1B	8238	0.99	80,000.00	8,000.00	8,000.00	80,000.00
sobutanol	78-83-1	FA1C3A	00439HD	0.99	80,000.00	8,000.00	8,000.00	80,000.00
l,4-Dioxane	123-91-1	RA1H3B	03053BD	0.99	80,000.00	8,000.00	8,000.00	80,000.00

Add + MVSC 74 18→20
L 75 1→7

CERTIFICATE OF COMPOSITION

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.: 558661 Lot No.: A042271

Description: Custom Volatiles Standard

Expiration Date[1]: July 2007 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Propanol (isopropanol)	67-63-0	99%	20000 ug/mL	+/- 0.1
2	1-Propanol	71-23-8	99%	20000 ug/mL	+/- 0.1
3	n-Hexane (C6)	110-54-3	99%	1000 ug/mL	+/- 0.1
4	Acetaldehyde dimethyl acetal	534-15-6	99%	5000 ug/mL	+/- 0.1
5	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	1000 ug/mL	+/- 0.1
6	tert-Amyl methyl ether (TAME)	994-05-8	99%	1000 ug/mL	+/- 0.1
7	n-Heptane (C7)	142-82-5	99%	1000 ug/mL	+/- 0.1
8	2-Chlorobenzotrifluoride	88-16-4	99%	1000 ug/mL	+/- 0.1
9	3-Chlorobenzotrifluoride	98-15-7	99%	1000 ug/mL	+/- 0.1
10	4-Chlorobenzotrifluoride	98-56-6	98%	1000 ug/mL	+/- 0.1
11	3-Chlorotoluene	108-41-8	99%	1000 ug/mL	+/- 0.1
12	1,2,3-Trimethylbenzene	526-73-8	99%	1000 ug/mL	+/- 0.1
13	Dicyclopentadiene	77-73-6	98%	1000 ug/mL	+/- 0.1
14	1,3,5-Trichlorobenzene	108-70-3	99%	1000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat #10921)

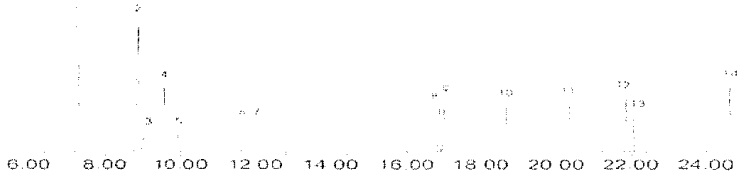
Carrier Gas:
helium @ 2.2 ml/min

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD



Manufactured By: FJT

John Liggett - QA 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 preperation with balance calibration verified using NISTtraceable weights (7 mass levels)
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

Table 14. BFB Standard

MVSC 3 1-710

RESTEK

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 Lot No.: A038650

Description: 4-Bromofluorobenzene Standard

Expiration Date[1]: January 2010 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)

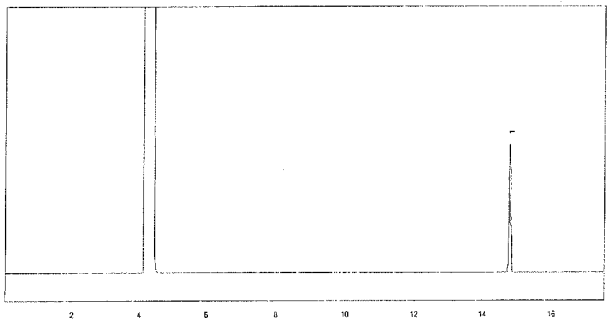
Carrier Gas:
hydrogen @ 40 cm/sec

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 Lidgett - Q.A. 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 preperation with balance calibration verified using NIST traceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

Page 1 1

Table 15. Internal Standard Mixture

MWSC08 (11-20)

RESTEK

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.: 30091 Lot No.: A036981

Description: L/C VOA Internal Standard Mix

Expiration Date[1]: April 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Difluorobenzene	540-36-3	99%	2500 ug/mL	+/- 0.1
2	Chlorobenzene-d5	3114-55-4	99%	2500 ug/mL	+/- 0.1
3	1,4-Dichlorobenzene-d4	3855-82-1	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)

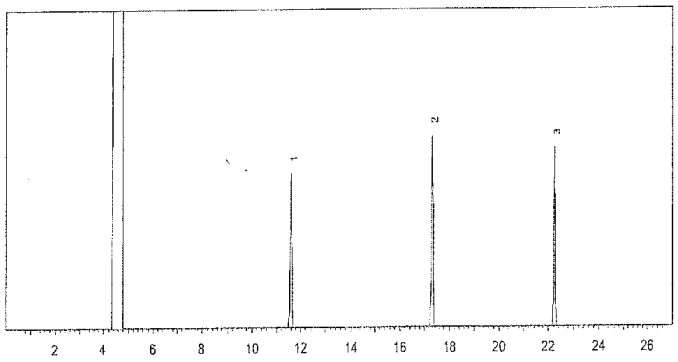
Carrier Gas:
hydrogen @ 40 cm/sec.

Temp. Program:
40°C (hold 2 min) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: n/a

John Lidgett - QA 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 addtion 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 preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

Page 1 1

Table 16. Surrogate Mixture

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Volatiles System Monitoring Spiking Solution

Product STM-262 **Page:** 1 of 1
Lot Number: CC-3176 **Lot Issue Date:** Oct-2006 **Expiration Date:** Nov-2009

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	CAS#	Analyte Lot	True Value
4-bromofluorobenzene	000460-00-4	12515BO	2511 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO5A-048	2504 ± 13 µg/mL
toluene-d8	002037-26-5	6D-549	2503 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

ACCREDITED
ISO 17025:2005 Accredited A2LA Cert. No. 0851.01
ISO 9001:2000 Registered TUV USA, Inc. Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

See Reverse For Additional Information

William J. Leahy
Quality Assurance Manager

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.

Title: Analytical Methods for GC/MS Semivolatile Samples by SW846 3rd Edition 8270C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

The analytical method is utilized for the analysis of water, air sampling media, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1. Table 1 includes CAS numbers and estimated quantitation limits for each analyte. Typical sample size should be 30 grams for soils and 1 liter for waters for large volume injection method 250mls. The method begins with the extraction of the sample aliquot either by sonication (soils) or separatory funnel extraction (waters), into 1:1 methylene chloride/ acetone mixture. The extraction volume is then concentrated to 1.0ml final volume for waters and soils. The extracts are prepared for analysis with the addition of internal standard to each vial. One microliter of each extract is then directly injected into a gas chromatograph and the compounds are separated by mass using a capillary column and analyzed using a mass spectrometer. A summary of the analysis procedure is provided in Attachment A.

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.

2.0 Summary of Method

See Scope and Application

3.0 Definitions

Additional definitions scan be found in the TAL Buffalo Laboratory Quality Manual (LQM)

4.0 Interferences

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.

4.1 See section 1.4 and 3.0 of method 8270C for other interferences, with the exception that there is no carryover in direct injection GCMS.

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

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 degreases 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 pipettes and pipette 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 Semi-volatiles column or equivalent

6.5.3 Mass Spectrometer
- HP5973
- Tuning compound PFTBA
- Scan Range 35-500 AMU/second

6.5.4 Data System
- HP Chemstation
- CHROM/TALS data analysis and LIMS 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
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.

7.2.2 Initial and Continuing Calibration Solutions
8270 Stock Solution

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc.	Final Conc. In Samples
CLP Semivo Calibration Mix	$MECL_2$	1000 ng/ul	400µl	2000ul	200 ng/ul	200 ug/L
Calibration Mix # 4	$MECL_2$	2000 ng/ul	200µl	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	$MECL_2$	5000 ng/ul	80µl	2000ul	200 ng/ul	200 ug/L
BN/AP Mix	$MECL_2$	4000 ng/ul	100µl	2000ul	200 ng/ul	200 ug/L
OLM Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzoic Acid	$MECL_2$	2000 ng/ul	400µl	2000ul	200 ng/ul	400 ug/L

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	***Solvent***	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. In Samples
Semivolatile 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

7.2.3.2.
Matrix Spiking Solution (11 compound)

The 11 compound matrix spiking solution consists of the following:

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

a. Using the Intermediate Acid and BN Standards, the Matrix Spike solution is prepared that contains each of the base-neutral compounds above at 100µg/ml in methanol and the acid compounds at 100µg/ml in methanol.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. in Solution	Final Conc. In Aqueous Samples
Acid Matrix Spike Intermediate BN Matrix Spike Intermediate	MeOH MeOH	10000ng/ul 5000ng/ul	5000uls 10000uls	500mls 500mls	100 ug/ml 100 ug/ml	100 µg/L 100 ug/L

7.2.3.3 Matrix Spiking Solution (all compound)

The all compound matrix spiking solution contains each of the following SVOA target analytes at 100µg/ml in methanol. Additional compounds may be included in the spike mixture if required for a specific project. For MCP/RCP work a specific list of client compounds will be used.

Ancenaphthene	Dibenzo(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Dibenzofuran	Isophorone
Anthracene	di-n-butyl phthalate	2-Methylnaphthalene
Benzo(a)anthracene	1,2-Dichlorobenzene	2-Methylphenol
Benzo(b)fluoranthene	1,3-Dichlorobenzene	4-Methylphenol
Benzo(k)fluoranthene	1,4-Dichlorobenzene	Naphthalene
Benzo(ghi)perylene	3,3'Dichlorobenzidine	2-Nitroaniline
Benzo(a)pyrene	2,4-Dichlorophenol	3- Nitroaniline
Benzoic acid	Diethyl phthalate	4- Nitroaniline
Benzyl alcohol	2,4-Dimethylphenol	Nitrobenzene
Bis(2-chloroethoxy)methane	Dimethyl phthalate	2-Nitrophenol
Bis(2-chloroethyl)ether	4,6-Dinitro-2-methylphenol	4-Nitrophenol
2,2'-oxybix(1-Chloropropane)	2,4-Dinitrophenol	N-nitrosodiphenylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-Di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	Pentachlorophenol
Butyl benzyl phthalate	Di-n-octyl phthalate	Phenanthrene
2-Chloroaniline	Fluoranthene	Phenol
4-Chloro-3-methylphenol	Fluorene	Pyrene
2-Chloronaphthalene	Hexachlorobenzene	1,2,4-Trichlorobenzene
2-Chlorophenol	Hexachlorobutadiene	2,4,5-Trichlorophenol
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
Chrysene	Hexachloroethane	Tetra Ethyl Lead
1,4 Dioxane		

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

8.1 Water samples may be collected in 1L (or more) amber glass containers with Teflonlined, screw-caps or 250ml bottles for LVI (Large Volume Injection).

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.

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 (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

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 LVI (Large Volume Injection) the sample will consist of 250ml of reagent water spike with 1ml of the surrogate spiking solution. 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.

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 is the problem, 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, 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 laboratory control sample, 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, laboratory control sample 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

9.2.4 If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, duplicate laboratory control samples (LCSD) may be processed. **For all MCP/RCP protocol work a LCSD must be used in each batch.**

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.

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

$$\text{Matrix Spike Recovery} = \frac{\text{SSR - SR}}{\text{SA}} x100$$

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

$$\text{RPD} = \frac{[\text{MSR - MSDR}]}{1/2\,(\text{MSR} + \text{MSDR})} \text{ X } 100$$

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 MSB:
The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for the matrix spike blank analysis also.

The matrix spike blank recovery limits are based on historical data and are updated annually.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analyses must be included with the report.

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

$$\%\ Recovery = \frac{Concentration\ (\vee\ amount)\ found}{Concentration\ (\vee\ amount)\ spiked}$$

Recovery limits for surrogate compounds are based on historical data and are updated annually.

9.3
Instrument QC

General Instrument Operating Conditions

- Gas Chromatograph; The following are recommended GC conditions that may vary slightly depending on the compound list and the column film thickness.
  Initial Temperature: 40-50°C
  Initial Hold Time: 3 minutes (hold time may vary to ensure proper chromatographic separation).
  Temperature program 40-50°C to 70°C at 20°C/min to 195 at
  16°C/min to 325 at 30°C/min
  Final Temperature: 325°C

Final Hold Time: As necessary for TCL compound identification
Injector Temperature: 250°C
Source Temperature: 230°C
Transfer Line Temperature: 310°C
Injector: splitless
Front Inlet Pressure: 7.00 psi
Purge Flow: 15.0 mL/min
Purge Time: 0.50 min
Total flow: 19.2 mL/min
Injection Volume: 1µl/5ul for LVI (Large Volume Injection)
Carrier Gas: Helium
Carrier Flow: 36 cm/sec

- Mass Spectrometer
  Electron Energy: 70 volts (nominal)
  Mass Range: 35 to 500 amu
  Scan Time: Not to exceed 1 second per scan

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 <5 and Benzidine <3). 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.

9.4.1 Technical Acceptance Criteria for the GC/MS Instrument Performance Check (DFTPP) is listed in Table 2.

9.4.2 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. **For all MCP/RCP work a separate low level calibration must be analyzed to meet client/project specific reporting limits.**

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 quadrapole 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

Five calibration standards are prepared which contain all target and surrogate compounds. A 20µl aliquot of internal standard solution is added to a 1mL aliquot of each calibration standard solution. The resulting concentration of internal standards is 40ng. A 1µl injection would result in a final concentration of 40ng on column. 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 3 and 6. Internal standard assignments are listed in Table 4.

Equation 4

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,
A_x = Area of the characteristic ion for the compound to be measured (see Table 4)
A_{is} = Area of the characteristic ion for specific internal standard (see Table 3)
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

$$\%RDS = \frac{Standard\ Deviation}{Mean}\ x\ 100$$

Where,

$$Standard\ Deviation = \sqrt{\frac{\sum_{i+1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

x_i = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

9.5.2 Acceptance Criteria for Initial Calibration

The average response factor (RRF) for each System Performance Check Compound (listed in Table 5) must be greater than or equal to the compound's minimum acceptable relative response factor of 0.050.

The %RSD over the initial calibration range for relative response factor for each Calibration Check (Table 5) compound %RSD must be less than or equal to the 30%.

The %RSD over the initial calibration range for the relative response factor for all other compounds must be less than or equal to 15%.

OR

A least squares regression correlation coefficient of greater than 0.990 for all compounds greater than 15% RSD.

OR

A non-linear coefficient of determination of greater than 0.990 for all compounds greater than 15% RSD. For a 2nd order non-linear regression, 6 calibration points must be used and for a 3rd order non-linear regression, 7 calibration points must be used.

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, a 80ng/µl aliquot of this independent standard is injected.

Recoveries of all compounds shall fall within ±20% of the expected value, however, recoveries of up to 40% are allowable for up to four compounds.

9.5.5 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance check may be analyzed and a 50ng/1µl standard may be analyzed to verify the calibration of the instrument.

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

The 50ng/µl standard is used for the continuing calibration. The relative response factor is calculated using procedures described for initial calibration.

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

$$\%\ Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} x\ 100$$

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:

$$\%Drift = \frac{Conc_E - Conc_A}{Conc_E} x100$$

Where:

$Conc_E$ = Expected Concentration
$Conc_A$ = Actual Concentration

9.5.7 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for each System Performance Check Compound must be greater than or equal 0.050.

The RRF of percent drift for Calibration Check Compounds must be less than 20%. The RRF percent difference or percent drift for all other compounds including TCL list compounds must be within ±20%, with up to four compounds within ±40%D. For expanded list and additional compounds not on the EPA TCL list a percent drift of 40% is allowed. Any analyte may have an elevated response >40%D if it is not detected in the associated samples, with the exception of APIX and priority pollutant compounds +/- 100%D.

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 or the response of any internal standard is outside of the-50%to +100% range, 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

9.6 Calibration Acceptance Summary

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method #8270				
Initial Cal	*Conc and # of stds*	*Type of Cal: Linear, …*		*How often performed?*
ICV	*80ng*	*LINEAR*	*+/- 20%*	*After initial cal.*
CCV	*50ng*	*LINEAR*	*+/- 20%*	

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:

Vol. Extract (ml) X 20 ul = 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.

10.2.1 Sample Log Book Entry

Samples are logged into the electronic instrument logbook prior to the start of the analysis.

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 that of the standard RRT. 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 ±20.0 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 30.0 and 70.0 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 judgement of the mass spectral interpretation specialist the identification is correct, the compound will be identified. Documentation of such by the specialist on the raw data is required.

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 $\pm$20 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 judgement 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 analyte was 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 $\pm$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 $\pm$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 with 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 Matrix Spike Blanks.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analyses must be included with the report.

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 4). The EICP area of primary characteristic ions of analytes listed in Tables 3 and 6 are used for quantitation.

In instances where manual quantitation 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

$$Concentration\ \mu g/L = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRFi)(V_o)(V_i)}$$

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_t = 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:

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

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

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRF_i)(V_i)(W_s)(D)}$$

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 µL)
V_i = Volume of the extract injected in microliters (µL)
D = $\frac{100 - \%\ moisture}{100}$
W_s = Weight of sample extracted in grams (g)
GPC= GPC factor (GPC = 2.0 to account for GCP 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:

$$\frac{\mu L \text{ most conc. Extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. Extract used to make dilution}}$$

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. **For all MCP/RCP protocol work TICS must be run on all samples identified as drinking water samples per the COC.**

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 Section xx 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.

12.5 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 market 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 III, Method 8270C, 12/96.

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: Relative Response Factor Criteria for ICV and CCV
17.5 Table 5: Characteristic Ions for Target Compounds and Surrogates
17.6 Table 6: Poor Performing Compounds
17.7 Attachment A: SOP Procedure Summary
17.8 Attachment C: Job Summary Checklist

18.0 Revision History

Revision 6, dated February 18, 2013

- Update Quality Assurance Manager, signature added
- Changed verbiage under GC run conditions

Revision 5, dated December 27, 2012

- In Section 9.5.4 and in the table for Section 9.6, ICV and CCV recoveries were changed to ±20% of the expected value from ±25%.
- Changed Quality Officer, signature added.

Revision 4, dated October 26, 2012

- 1.1 Large Volume Injection reference
- 6.5.2 Changed column vendor to Phenomenex
- 8.1 Large Volume Injection sample size reference
- 9.1.1 LVI (Large Volume Injection) MBLK criteria
- 9.3 Injection volume change under instrument conditions for LVI
- 9.2 Changed MSB references to LCS
- 9.2.4 Added LCSD requirement for MCP/RCP work
- 9.2.10 Removal of AFCEE/ACE references
- 9.3 Injection volume change under instrument conditions for LVI
- 9.5 Added ICAL requirements for RCP/MCP work
- 11.4 Added MCP/RCP TIC requirement for drinking waters

Revision 3, dated January 12, 2012

- Changed Quality Manager, signature added.
- Removed all references to Army Corp of Engineers and AFCEE
- Removed all Element data processing references
- Removed all manual logbook references
- Added Chrom and TALS references throughout as needed
- Added analytes to Table 1.
- Added new Summary sheet

Revision 2, dated February 01, 2010

- Removed AFCEE attachment
- Removed ACOE attachment
- Added log book copy attachments, referenced in section 10.2.1
- Addition of 69 ion criteria to table
- Section 11.2 and 11.3 updated to state that the relative response factor is taken from the initial calibration
- Equations in section 11.2 and 11.3 were updated to reflect correct subscript for (RRF) to (RRFi) and for (Vc) to (Vt) and for (I3) to (Is)
- Updated attachment 1 to include Element and deleted AIMS reference
- Added APIX ,TCL list and priority pollutant %D statement in section 9.5.7
- Added Table 6. Poor Performers

Revision 1, dated June 10, 2009

- Removal of grand mean reference
- Integration for TestAmerica and STL operation
- Change to QA Manager, signature updated
- Change to Department Manager, signature updated

TABLE 1

Semivolatiles Target Compound List and Contract Estimated Quantitation Limits

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
29.	1,3-Dinitrobenzene	99-65-0	10	330
30.	Thionazin	297-97-2	10	330
31.	N-Nitrosomethylethylamine	10595-95-6	10	330
32.	1,2-Diphenylhydrazine	122-66-7	10	330
33.	2-Acetylaminofluorene	53-96-3	10	330
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-chloroisopropl)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-Nitroanline	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

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
71.	Diethlphthalate	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
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

TABLE 2

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30.0 - 60.0 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	40.0 - 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see Note)
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	Greater than 1.0% of than mass 198
441	Present but less than mass 443
442	40.0 – 110.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

Note: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be greater to 110 percent that of m/z 198.

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-Bromophenylphenolether	3,3'-Dichlorobenzidine	Benzo(a)phyrne
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloroaphthalene	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	Carbzole	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-Chloroanaline	Acenaphthene	Di-n-butylphthalate		
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitorphenol	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-Nitrosobutyl-amine	4-Chlorophenyl-phenylether			
Benzaldehyde	Caprolactam	Fluorene			
Acetophenone	1,2,4,5-Tetrachlorbenz.	4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			
		1,1'-Biphenyl			
		2,3,4,6-Tetrachlorophenol			

TABLE 4

Relative Response Factor Criteria for Initial and Continuing Calibration of Semivolatile Target Compounds and Surrogates

Semivolatile Compounds	Minimum RRF	Maximum % RSD	Maximum % Diff
Acenaphthene (CCC)	none	30	±20
1,4-Dichlorobenzene (CCC)	none	30	±20
Hexachlorobutadiene (CCC)	none	30	±20
N-Nitrosodiphenylamine (CCC)	none	30	±20
Di-n-octylphthalate (CCC)	none	30	±20
Flouranthene (CCC)	none	30	±20
Benzo(a)pyrene (CCC)	none	30	±20
4-Chloro-3-methylphenol (CCC)	none	30	$^{+}$20
2,4-Dichlorophenol (CCC)	none	30	±20
2-Nitrophenol (CCC)	none	30	±20
Phenol (CCC)	none	30	±20
Pentachlorophenol(CCC)	none	30	±20
2,4,6-Trichlorophenol (CCC)	none	30	±20
N-Nitroso-di-n-propylamine (SPCC)	0.050	None	none
Hexachlorocyclopentadiene (SPCC)	0.050	None	none
2,4-Dinitrophenol (SPCC)	0.050	None	none
4-Nitrophenol (SPCC)	0.050	None	none

Table 5

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
Dimethylphthalate	163	194, 164

Parameters	Primary Quantitation Ion	Secondary Ion(s)
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
Benzo(a)pyrene	252	253, 125

Parameters	Primary Quantitation Ion	Secondary Ion(s)
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-Tribormophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

Table 6: Poor Performing Compounds

1,4 dioxane	N-nitrosodimethylamine
Pyridine	Methane sulfanate
Benzaldehyde	1-napthylamine
2-napthylamine	N-nitrosodiphenylamine
N-nitrosopiperidine	N-nitrosomorpholine
N-nitrosopyrrolidine	p-Dimethylamino azobenzene
p-phenylenediamine	a,a-dimethylphenethylamine
Methapyriline	Aniline
4-Chloroaniline	2-nitroaniline
3-nitroaniline	4-nitoraniline
2-Picoline	3,3- dimethylbenzidine
3,3- dichlorobenzidine	Benzidine
Benzaldehyde	Benzoic acid
2,4- dinitrophenol	4-nitrophenol
Dinoseb	Hexachlorophene
Hexachlorocyclopentadiene	o,o,o-triethylphosphoro-thioate
Kepone	Phthalic Anhydride
Tetra Ethyl Lead	2,6 dinitrotolulene
4,6- dinitro-2-methylphenol	Famphur
Caprolactum	Pentachlorophenol
Simazine	1,3,5 trinitrobenzene
4-nitroquinoline-1-oxide	Tri-cresylphosphate
(2)-9- octadecanamide	N,N Dimethylacetamide

*****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%.

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 3rd 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 (CCC)
   2. Evaluate the continuing; 4pts may be out but none over 40%d.
   3. If CCC does not pass criteria, then a 5pt. curve (ICC) must be shot.

IV. Load Samples;
   1. Load blanks and MSBs 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. Upload tunes, ICC's and CCC's.
   2. Enter all samples to be included with the job.
   3. Calculate, close and run batch historical check

VII. Review Data;
   1. Correct or explain any errors on the batch historical check
   2. Check that all calculations have been made correctly.
   3. Turn in job for validation.

Attachment C: Job Summary Checklist

TESTAMERICA BUFFALO GCMS SV CHECKLIST - METHODS 8270C

Work Order ID: ______ TALS Calibration Batch ID: ______

Instrument ID: ______ TALS Curve ID: ______

Analysis Date: ______ TALS Batch ID: ______

Chrom Worklist ID: ______

1st level reviewer/date: ______ Prep Batch ID: ______

QAPP #: ______

2nd level reviewer/date: ______

I agree that I have reviewed the data as indicated on this checklist

	1st level (Y/N)	2nd level (Y/N)
1: DFTPP Tune		
The Tune documentation is present and meets method criteria	☐	☐
Samples analyzed within 12 hour tune window	☐	☐
Tune time: ______		
Comments: ______		
2: CCV Std.		
The SPCC meet criteria (RRF) - greater than the minimum	☐	☐
SPCC are N-Nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol (avg RF ≥ 0.050).		
Comments: ______		
The CCC meet criteria (%RSD or % Drift ≤20%)	☐	☐
CCCs are Phenol, N-Nitrosodiphenylamine, Hexachlorobutadiene, Fluoranthene, Di-n-octylphthalate, Benzo(a)pyrene, Acenaphthene, 4-chloro-3-methylphenol, 2-Nitrophenol, 2,4-Dichlorophenol, 2,4,6-Trichlorophenol, 1,4-dichlorobenzene		
Comments: ______		
All other Compounds %RSD criteria must be ≤25% *(%RSD or % Drift) (or as specified by program or SOP)*	☐	☐
Comments/Outliers: ______		
4 analytes or less out by less than 40%	☐	☐
Internal standard 50-200% of ICAL	☐	☐
Correct CAL linked to samples	☐	☐
ICV linked to samples	☐	☐
Retention time windows updated and correct	☐	☐
3: QC Criteria		
Method Blank analyzed, results < RL or QC specific program	☐	☐
LCS and MS/MSD analyzed, meets criteria	☐	☐
Document and qualify QC outliers: ______		
4: Sample Results Summary (samples, mb, spikes)		
Surrogate results within QC criteria	☐	☐
Document and qualify Outliers: ______		
Manual integrations properly documented (before/after chromatograms)	☐	☐
All analytes within calibration range	☐	☐
TICs reviewed if required	☐	☐
All samples extracted and analyzed within required hold time	☐	☐
Samples submitted for reanalysis and/or re-extraction	☐	☐
Sample re-analysis compared to original dilution/analysis	☐	☐
Original sample analysis files or batch number		
Historical data check run ______		☐
Job Exceptions ______		

Additional Comments: ______

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WADSWORTH CENTER

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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

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MR. CHRISTOPHER SPENCER
TESTAMERICA BUFFALO
10 HAZELWOOD DRIVE - SUITE 106
AMHERST, NY 14228

NY Lab Id No: 10026

Is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Dissolved Gases	
Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175
Drinking Water Metals I	
Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Drinking Water Metals II	
Aluminum, Total	EPA 200.7 Rev. 4.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Drinking Water Metals III	
Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4
Drinking Water Miscellaneous	
Endothall	EPA 548.1
Methyl Iodide	EPA 524.2
Organic Carbon, Total	SM 19-22 5310D (-00)
Turbidity	EPA 180.1 Rev. 2.0
Drinking Water Non-Metals	
Alkalinity	EPA 310.2

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Drinking Water Non-Metals	
Alkalinity	SM 18-22 2320B (-97)
Calcium Hardness	EPA 200.7 Rev. 4.4
	SM 18-22 2340B (-97)
Chloride	EPA 300.0 Rev. 2.1
	SM 18-22 4110B (-00)
	SM 21-22 4500-Cl- E (-97)
Color	SM 18-22 2120B (-01)
Cyanide	SM 18-22 4500-CN E (-99)
	EPA 335.4 Rev. 1.0
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 18-22 4110B (-00)
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
	SM 18-22 4110B (-00)
Nitrite (as N)	EPA 353.2 Rev. 2.0
Orthophosphate (as P)	SM 18-22 4500-P E (-99)
Solids, Total Dissolved	SM 18-22 2540C (-97)
Specific Conductance	EPA 120.1 Rev. 1982
Sulfate (as SO4)	ASTM D516-90 02 & 07
	EPA 300.0 Rev. 2.1
	SM 18-22 4110B (-00)
Drinking Water Trihalomethanes	
Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2

Drinking Water Trihalomethanes	
Dibromochloromethane	EPA 524.2
Total Trihalomethanes	EPA 524.2
Fuel Additives	
Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2
Microextractibles	
1,2-Dibromo-3-chloropropane	EPA 504.1
1,2-Dibromoethane	EPA 504.1
Volatile Aromatics	
1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2

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Volatile Aromatics	
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dibromo-3-chloropropane	EPA 524.2
1,2-Dibromoethane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2

Volatile Halocarbons	
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

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All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
	EPA 624
Acrylonitrile	EPA 8260C
	EPA 624
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1,2-Diphenylhydrazine	EPA 8270D
1,4-Phenylenediamine	EPA 8270D
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 625
	EPA 8270D
Carbazole	EPA 625
	EPA 8270D
Diphenylamine	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Propionitrile	EPA 8260C

Amines	
Pyridine	EPA 625
	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 625
	EPA 8270D
3,3'-Dimethylbenzidine	EPA 8270D
Benzidine	EPA 625
	EPA 8270D
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081B
	EPA 608
4,4'-DDE	EPA 8081B
	EPA 608
4,4'-DDT	EPA 8081B
	EPA 608
Aldrin	EPA 8081B
	EPA 608
alpha-BHC	EPA 8081B
	EPA 608
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
	EPA 608
Chlordane Total	EPA 8081B
	EPA 608
Chlorobenzilate	EPA 8270D

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Chlorinated Hydrocarbon Pesticides	
delta-BHC	EPA 8081B
	EPA 608
Diallate	EPA 8270D
Dieldrin	EPA 8081B
	EPA 608
Endosulfan I	EPA 8081B
	EPA 608
Endosulfan II	EPA 8081B
	EPA 608
Endosulfan sulfate	EPA 8081B
	EPA 608
Endrin	EPA 8081B
	EPA 608
Endrin aldehyde	EPA 8081B
	EPA 608
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
	EPA 608
Heptachlor epoxide	EPA 8081B
	EPA 608
Isodrin	EPA 8270D
Kepone	EPA 8270D
Lindane	EPA 8081B
	EPA 608
Methoxychlor	EPA 8081B

Chlorinated Hydrocarbon Pesticides	
Methoxychlor	EPA 608
Mirex	EPA 8081B
	SM 6630C-00
PCNB	EPA 8270D
Toxaphene	EPA 8081B
	EPA 608
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 625
	EPA 8270D
2-Chloronaphthalene	EPA 625
	EPA 8270D
Hexachlorobenzene	EPA 625
	EPA 8270D
Hexachlorobutadiene	EPA 625
	EPA 8270D
Hexachlorocyclopentadiene	EPA 625
	EPA 8270D
Hexachloroethane	EPA 625
	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A

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WADSWORTH CENTER

Expires 12:01 AM April 01, 2015
Issued April 01, 2014

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. CHRISTOPHER SPENCER
TESTAMERICA BUFFALO
10 HAZELWOOD DRIVE - SUITE 106
AMHERST, NY 14228

NY Lab Id No: 10026

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorophenoxy Acid Pesticides	
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
Dalapon	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
Demand	
Biochemical Oxygen Demand	SM 5210B-01,-11
Carbonaceous BOD	SM 5210B-01,-11
Chemical Oxygen Demand	HACH 8000
	EPA 410.4 Rev. 2.0
Dissolved Gases	
Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175
Fuel Oxygenates	
Di-isopropyl ether	EPA 8260C
Ethanol	EPA 8015D
Methyl tert-butyl ether	EPA 8260C
	EPA 8021B
tert-amyl methyl ether (TAME)	EPA 8260C
tert-butyl alcohol	EPA 8260C
	EPA 8015D
Fuel Oxygenates	
tert-butyl ethyl ether (ETBE)	EPA 8260C
Haloethers	
4-Bromophenylphenyl ether	EPA 625
	EPA 8270D
4-Chlorophenylphenyl ether	EPA 625
	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 625
	EPA 8270D
Bis(2-chloroethyl)ether	EPA 625
	EPA 8270D
Bis(2-chloroisopropyl) ether	EPA 625
	EPA 8270D
Mineral	
Alkalinity	EPA 310.2
	SM 2320B-97,-11
Calcium Hardness	EPA 200.7 Rev. 4.4
Chloride	EPA 300.0 Rev. 2.1
	SM 4110B-00,-11
	SM 4500-Cl- E-97,-11
	EPA 9056A
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 4110B-00,-11
	SM 4500-F C-97,-11
	EPA 9056A
Hardness, Total	SM 2340C-97,-11

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Mineral		**Nitrosoamines**	
Hardness, Total	SM 2340B-97,-11	N-Nitrosodiphenylamine	EPA 8270D
Sulfate (as SO4)	ASTM D516-90 02 & 07	N-nitrosomethylethylamine	EPA 8270D
	EPA 300.0 Rev. 2.1	N-nitrosomorpholine	EPA 8270D
	SM 4110B-00,-11	N-nitrosopiperidine	EPA 8270D
	EPA 9056A	N-Nitrosopyrrolidine	EPA 8270D
Nitroaromatics and Isophorone		**Nutrient**	
1,3,5-Trinitrobenzene	EPA 8270D	Ammonia (as N)	EPA 350.1 Rev. 2.0
1,3-Dinitrobenzene	EPA 8270D	Kjeldahl Nitrogen, Total	EPA 351.2 Rev. 2.0
1,4-Naphthoquinone	EPA 8270D	Nitrate (as N)	EPA 353.2 Rev. 2.0
2,4-Dinitrotoluene	EPA 625		EPA 300.0 Rev. 2.1
	EPA 8270D		SM 4110B-00,-11
2,6-Dinitrotoluene	EPA 625		SM 4500-NO3 F-00,-11
	EPA 8270D		EPA 9056A
Isophorone	EPA 625	Nitrite (as N)	EPA 353.2 Rev. 2.0
	EPA 8270D		SM 4500-NO3 F-00,-11
Nitrobenzene	EPA 625	Orthophosphate (as P)	SM 4500-P E-99,-11
	EPA 8270D	Phosphorus, Total	SM 4500-P E-99,-11
Nitrosoamines		**Organophosphate Pesticides**	
N-Nitrosodiethylamine	EPA 8270D	Atrazine	EPA 8270D
N-Nitrosodimethylamine	EPA 625	Dimethoate	EPA 8270D
	EPA 8270D	Disulfoton	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D	Famphur	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 625	Parathion ethyl	EPA 8270D
	EPA 8270D	Parathion methyl	EPA 8270D
N-Nitrosodiphenylamine	EPA 625	Phorate	EPA 8270D

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Organophosphate Pesticides	
Simazine	EPA 8270D
Thionazin	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Phthalate Esters	
Benzyl butyl phthalate	EPA 625
	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270D
Diethyl phthalate	EPA 625
	EPA 8270D
Dimethyl phthalate	EPA 625
	EPA 8270D
Di-n-butyl phthalate	EPA 625
	EPA 8270D
Di-n-octyl phthalate	EPA 625
	EPA 8270D
Polychlorinated Biphenyls	
PCB-1016	EPA 8082A
	EPA 608
PCB-1221	EPA 8082A
	EPA 608
PCB-1232	EPA 8082A
	EPA 608
PCB-1242	EPA 8082A
	EPA 608
PCB-1248	EPA 8082A
	EPA 608
PCB-1254	EPA 8082A
	EPA 608
PCB-1260	EPA 8082A
	EPA 608
PCB-1262	EPA 8082A
PCB-1268	EPA 8082A
Polynuclear Aromatics	
2-Acetylaminofluorene	EPA 8270D
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 625
	EPA 8270D
Acenaphthylene	EPA 625
	EPA 8270D
Anthracene	EPA 625
	EPA 8270D
Benzo(a)anthracene	EPA 625
	EPA 8270D
Benzo(a)pyrene	EPA 625
	EPA 8270D

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Polynuclear Aromatics

Benzo(b)fluoranthene	EPA 625
	EPA 8270D
Benzo(ghi)perylene	EPA 625
	EPA 8270D
Benzo(k)fluoranthene	EPA 625
	EPA 8270D
Chrysene	EPA 625
	EPA 8270D
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270D
Fluoranthene	EPA 625
	EPA 8270D
Fluorene	EPA 625
	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270D
Naphthalene	EPA 625
	EPA 8270D
Phenanthrene	EPA 625
	EPA 8270D
Pyrene	EPA 625
	EPA 8270D

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 625

Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 625
	EPA 8270D
2,4-Dichlorophenol	EPA 625
	EPA 8270D
2,4-Dimethylphenol	EPA 625
	EPA 8270D
2,4-Dinitrophenol	EPA 625
	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 625
	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 625
	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 625
	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 625
	EPA 8270D
Cresols, Total	EPA 625
	EPA 8270D
Pentachlorophenol	EPA 8151A

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Priority Pollutant Phenols	
Pentachlorophenol	EPA 625
	EPA 8270D
Phenol	EPA 625
	EPA 8270D
Residue	
Settleable Solids	SM 2540 F-97,-11
Solids, Total	SM 2540 B-97,-11
Solids, Total Dissolved	SM 2540 C-97,-11
Solids, Total Suspended	SM 2540 D-97,-11
Semi-Volatile Organics	
1,1'-Biphenyl	EPA 8270D
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 625
	EPA 8270D
Benzaldehyde	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Caprolactam	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Semi-Volatile Organics	
Methyl methanesulfonate	EPA 8270D
n-Decane	EPA 625
n-Octadecane	EPA 625
O,O,O-Triethyl phosphorothioate	EPA 8270D
p-Dimethylaminoazobenzene	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
	EPA 8021B
1,2-Dichlorobenzene	EPA 8260C
	EPA 624
1,3,5-Trimethylbenzene	EPA 8260C
	EPA 8021B
1,3-Dichlorobenzene	EPA 8260C
	EPA 624
1,4-Dichlorobenzene	EPA 8260C
	EPA 624
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
	EPA 8021B
	EPA 624
	EPA 602

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Volatile Aromatics	
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
	EPA 624
Ethyl benzene	EPA 8260C
	EPA 8021B
	EPA 624
	EPA 602
Isopropylbenzene	EPA 8260C
	EPA 8021B
m/p-Xylenes	EPA 8260C
	EPA 624
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
	EPA 8021B
n-Propylbenzene	EPA 8260C
	EPA 8021B
o-Xylene	EPA 8260C
	EPA 624
p-Isopropyltoluene (P-Cymene)	EPA 8260C
	EPA 8021B
sec-Butylbenzene	EPA 8260C
	EPA 8021B
Styrene	EPA 8260C
	EPA 624
tert-Butylbenzene	EPA 8260C
Toluene	EPA 8260C

Volatile Aromatics	
Toluene	EPA 8021B
	EPA 624
	EPA 602
Total Xylenes	EPA 8260C
	EPA 8021B
	EPA 624
	EPA 602
Volatile Chlorinated Organics	
Epichlorohydrin	EPA 8260C
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
	EPA 624
1,1,2,2-Tetrachloroethane	EPA 8260C
	EPA 624
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
	EPA 624
1,1-Dichloroethane	EPA 8260C
	EPA 624
1,1-Dichloroethene	EPA 8260C
	EPA 624
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C

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Volatile Halocarbons	
1,2-Dibromo-3-chloropropane	EPA 8011
1,2-Dibromoethane	EPA 8260C
	EPA 8011
1,2-Dichloroethane	EPA 8260C
	EPA 624
1,2-Dichloropropane	EPA 8260C
	EPA 624
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
	EPA 624
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
	EPA 624
Bromoform	EPA 8260C
	EPA 624
Bromomethane	EPA 8260C
	EPA 624
Carbon tetrachloride	EPA 8260C
	EPA 624
Chloroethane	EPA 8260C
	EPA 624
Chloroform	EPA 8260C
	EPA 624

Volatile Halocarbons	
Chloromethane	EPA 8260C
	EPA 624
cis-1,2-Dichloroethene	EPA 8260C
	EPA 624
cis-1,3-Dichloropropene	EPA 8260C
	EPA 624
Dibromochloromethane	EPA 8260C
	EPA 624
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
	EPA 624
Hexachlorobutadiene, Volatile	EPA 8260C
Methyl Iodide	EPA 8260C
Methylene chloride	EPA 8260C
	EPA 624
Tetrachloroethene	EPA 8260C
	EPA 624
trans-1,2-Dichloroethene	EPA 8260C
	EPA 624
trans-1,3-Dichloropropene	EPA 8260C
	EPA 624
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
	EPA 624
Trichlorofluoromethane	EPA 8260C
	EPA 624

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Volatile Halocarbons	
Vinyl chloride	EPA 8260C
	EPA 624
Volatiles Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
2-Nitropropane	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C
Cyclohexane	EPA 8260C
Ethyl Acetate	EPA 8260C
Ethylene Glycol	EPA 8260C
	EPA 8015D
Isobutyl alcohol	EPA 8260C
	EPA 8015D
Methyl acetate	EPA 8260C
Methyl cyclohexane	EPA 8260C
	EPA 8015D
Vinyl acetate	EPA 8260C
Wastewater Metals I	
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Lead, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Magnesium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A

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Wastewater Metals I		**Wastewater Metals II**	
Manganese, Total	EPA 200.8 Rev. 5.4	Arsenic, Total	EPA 6010C
Nickel, Total	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C		EPA 200.8 Rev. 5.4
	EPA 6020A	Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4		EPA 6010C
Potassium, Total	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C		EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4	Chromium VI	EPA 7196A
	EPA 6010C		SM 3500-Cr B-09,-11
	EPA 6020A	Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 200.8 Rev. 5.4		EPA 7470A
Sodium, Total	EPA 200.7 Rev. 4.4	Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C		EPA 6010C
Strontium, Total	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C		EPA 200.8 Rev. 5.4
	EPA 6020A	Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4		EPA 6010C
Wastewater Metals II			EPA 6020A
Aluminum, Total	EPA 200.7 Rev. 4.4		EPA 200.8 Rev. 5.4
	EPA 6010C	Zinc, Total	EPA 200.7 Rev. 4.4
Antimony, Total	EPA 200.7 Rev. 4.4		EPA 6010C
	EPA 6010C		EPA 6020A
	EPA 6020A		EPA 200.8 Rev. 5.4
	EPA 200.8 Rev. 5.4	**Wastewater Metals III**	
Arsenic, Total	EPA 200.7 Rev. 4.4	Cobalt, Total	EPA 200.7 Rev. 4.4

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2015
Issued April 01, 2014

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. CHRISTOPHER SPENCER
TESTAMERICA BUFFALO
10 HAZELWOOD DRIVE - SUITE 106
AMHERST, NY 14228

NY Lab Id No: 10026

Is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Wastewater Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Tin, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Titanium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Wastewater Miscellaneous	
Boron, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Bromide	EPA 300.0 Rev. 2.1
	SM 4110B-00,-11
	EPA 9056A
Color	SM 2120B-01,-11
Cyanide, Total	LACHAT 10-204-00-1-X
	SM 4500-CN E-99,-11
	EPA 335.4 Rev. 1.0

Wastewater Miscellaneous	
Cyanide, Total	EPA 9012B
Oil and Grease Total Recoverable (HEM	EPA 1664A
Organic Carbon, Total	SM 5310D-00,-11
	EPA 9060A
Phenols	EPA 420.4 Rev. 1.0
	EPA 9065
	EPA 9066
Specific Conductance	EPA 120.1 Rev. 1982
	SM 2510B-97,-11
	EPA 9050A
Sulfide (as S)	SM 4500-S2- F-00,-11
	SM 4500-S2- D-00,-11
Surfactant (MBAS)	SM 5540C-00,-11
Total Organic Halides	EPA 9020B
Total Petroleum Hydrocarbons	EPA 1664A
Turbidity	EPA 180.1 Rev. 2.0
Sample Preparation Methods	
	SM 4500-P B(5)-99,-11
	EPA 5030C
	EPA 200.2
	EPA 3010A
	EPA 3005A
	EPA 3510C
	EPA 3520C
	EPA 3020A

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All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1,2-Diphenylhydrazine	EPA 8270D
1,4-Phenylenediamine	EPA 8270D
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Diphenylamine	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 8270D
3,3'-Dimethylbenzidine	EPA 8270D
Benzidine	EPA 8270D

Characteristic Testing	
Corrosivity	EPA 9040C
	EPA 9045D
Free Liquids	EPA 9095B
Ignitability	EPA 1010A
Reactivity	SW-846 Ch7 Sec. 7.3
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311
Chlorinated Hydrocarbon Pesticides	
2,4'-DDD (Mitotane)	EPA 8081B
4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
Atrazine	EPA 8270D
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
Chlorobenzilate	EPA 8270D
delta-BHC	EPA 8081B
Diallate	EPA 8270D
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B

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Chlorinated Hydrocarbon Pesticides	
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Kepone	EPA 8270D
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Pentachloronitrobenzene	EPA 8270D
Toxaphene	EPA 8081B
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Hexachlorophene	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D

Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
Dalapon	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
Pentachlorophenol	EPA 8151A
Haloethers	
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Bis(2-chloroisopropyl) ether	EPA 8270D
Metals I	
Barium, Total	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 6010C
	EPA 6020A
Calcium, Total	EPA 6010C
Chromium, Total	EPA 6010C
	EPA 6020A
Copper, Total	EPA 6010C
	EPA 6020A
Iron, Total	EPA 6010C
Lead, Total	EPA 6010C

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All approved analytes are listed below:

Metals I	
Lead, Total	EPA 6020A
Magnesium, Total	EPA 6010C
Manganese, Total	EPA 6010C
	EPA 6020A
Nickel, Total	EPA 6010C
	EPA 6020A
Potassium, Total	EPA 6010C
Silver, Total	EPA 6010C
	EPA 6020A
Sodium, Total	EPA 6010C
Strontium, Total	EPA 6010C
Metals II	
Aluminum, Total	EPA 6010C
Antimony, Total	EPA 6010C
	EPA 6020A
Arsenic, Total	EPA 6010C
	EPA 6020A
Beryllium, Total	EPA 6010C
	EPA 6020A
Lithium, Total	EPA 6010C
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010C
	EPA 6020A
Vanadium, Total	EPA 6010C
Zinc, Total	EPA 6010C

Metals II	
Zinc, Total	EPA 6020A
Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
Molybdenum, Total	EPA 6010C
	EPA 6020A
Thallium, Total	EPA 6010C
	EPA 6020A
Tin, Total	EPA 6010C
Titanium, Total	EPA 6010C
Minerals	
Bromide	EPA 9056A
Chloride	EPA 9251
	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO4)	EPA 9038
	EPA 9056A
Miscellaneous	
Boron, Total	EPA 6010C
Cyanide, Total	EPA 9012B
Organic Carbon, Total	EPA 9060A
Phenols	EPA 9066
Specific Conductance	EPA 9050A

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All approved analytes are listed below:

Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
1,3-Dinitrobenzene	EPA 8270D
1,4-Dinitrobenzene	EPA 8270D
1,4-Naphthoquinone	EPA 8270D
2,4-Dinitrotoluene	EPA 8270D
2,6-Dinitrotoluene	EPA 8270D
4-Dimethylaminoazobenzene	EPA 8270D
Hydroquinone	EPA 8270D
Isophorone	EPA 8270D
Nitrobenzene	EPA 8270D
Pyridine	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrients	
Nitrate (as N)	EPA 9056A
Organophosphate Pesticides	
Dimethoate	EPA 8270D
Disulfoton	EPA 8270D
Famphur	EPA 8270D
Parathion ethyl	EPA 8270D
Parathion methyl	EPA 8270D
Phorate	EPA 8270D
Sulfotepp	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polychlorinated Biphenyls	
PCB-1016	EPA 8082A
PCB-1221	EPA 8082A
PCB-1232	EPA 8082A
PCB-1242	EPA 8082A
PCB-1248	EPA 8082A
PCB-1254	EPA 8082A

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Polychlorinated Biphenyls	
PCB-1260	EPA 8082A
PCB-1262	EPA 8082A
PCB-1268	EPA 8082A
Polynuclear Aromatic Hydrocarbons	
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(ghi)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D
Dibenzo(a,e)pyrene	EPA 8270D
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D
2,4-Dichlorophenol	EPA 8270D
2,4-Dimethylphenol	EPA 8270D
2,4-Dinitrophenol	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 8270D
Pentachlorophenol	EPA 8270D
Phenol	EPA 8270D
Semi-Volatile Organics	
1,1'-Biphenyl	EPA 8270D
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 8270D
Benzaldehyde	EPA 8270D

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All approved analytes are listed below:

Semi-Volatile Organics	
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Caprolactam	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
O,O,O-Triethyl phosphorothioate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
	EPA 8021B
1,2-Dichlorobenzene	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
	EPA 8021B
1,3-Dichlorobenzene	EPA 8260C
1,4-Dichlorobenzene	EPA 8260C
2-Chlorotoluene	EPA 8260C
	EPA 8021B
4-Chlorotoluene	EPA 8260C
	EPA 8021B
Benzene	EPA 8260C
	EPA 8021B
Volatile Aromatics	
Bromobenzene	EPA 8260C
	EPA 8021B
Chlorobenzene	EPA 8260C
Ethyl benzene	EPA 8260C
	EPA 8021B
Isopropylbenzene	EPA 8260C
	EPA 8021B
m/p-Xylenes	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
	EPA 8021B
n-Propylbenzene	EPA 8260C
	EPA 8021B
o-Xylene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
	EPA 8021B
sec-Butylbenzene	EPA 8260C
	EPA 8021B
Styrene	EPA 8260C
tert-Butylbenzene	EPA 8260C
	EPA 8021B
Toluene	EPA 8260C
	EPA 8021B
Total Xylenes	EPA 8260C
	EPA 8021B

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Volatile Chlorinated Organics	
Epichlorohydrin	EPA 8260C
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260C
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C
1,1-Dichloroethene	EPA 8260C
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Bromomethane	EPA 8260C
Carbon tetrachloride	EPA 8260C

Volatile Halocarbons	
Chloroethane	EPA 8260C
Chloroform	EPA 8260C
Chloromethane	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromochloromethane	EPA 8260C
Dibromomethane	EPA 8260C
	EPA 8021B
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methyl iodide	EPA 8260C
Methylene chloride	EPA 8260C
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,3-Dichloropropene	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatile Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
2-Nitropropane	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C

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All approved analytes are listed below:

Volatile Organics	
Acetone	EPA 8260C
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C
Cyclohexane	EPA 8260C
Ethyl Acetate	EPA 8260C
Ethylene Glycol	EPA 8015D
Isobutyl alcohol	EPA 8260C
	EPA 8015D
Methyl acetate	EPA 8260C
Methyl cyclohexane	EPA 8260C
Methyl tert-butyl ether	EPA 8260C
Propionitrile	EPA 8260C
tert-butyl alcohol	EPA 8015D
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5035A-L
	EPA 5035A-H
	EPA 3580A
	EPA 3010A
	EPA 3005A
	EPA 3050B
	EPA 3550C
	EPA 3020A
	EPA 3546

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All approved analytes are listed below:

Polynuclear Aromatics	
Benzo(a)pyrene	NIOSH 5515
Naphthalene	NIOSH 5515
Purgeable Aromatics	
Benzene	NIOSH 1501
Ethyl benzene	NIOSH 1501
m/p-Xylenes	NIOSH 1501
o-Xylene	NIOSH 1501
Toluene	NIOSH 1501
Total Xylenes	NIOSH 1501

Serial No.: 50094

APPENDIX D

STANDARD OPERATING PROCEDURES (SOPS)

DECONTAMINATION
STANDARD OPERATING PROCEDURE D.1

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is generally applicable to the development and application of a decontamination program for a field investigation program in level D health and safety protection.

2.0 Summary of Method

2.1 This document has been prepared to assist personnel with the performance of specific tasks and procedures related to decontamination procedures during implementation of certain investigation activities. The procedures addressed in this SOP include the following:

- Personnel Decontamination Procedures
- Decontamination of Drilling Equipment
- Decontamination of Sampling Equipment
- Decontamination of Support Equipment
- Management of Investigation Derived Waste (IDW)

3.0 Health and Safety Issues

3.1 As with any activities associated with potential contaminants, work tasks should be conducted in strict accordance with Environmental Protection Agency (EPA), Occupation Safety & Health Administration (OSHA), client, and WESTON safety policy and procedures. This should include preparation of a site-specific Health and Safety Plan (HASP) to ensure that all aspects of potential risk are evaluated and properly addressed. A HASP has been prepared for the Study Area (see Appendix A of the Work Plan).

4.0 Personnel Qualifications

4.1 All field personnel with potential for exposure to contaminated media are required to take the 40-hour Health and Safety Training and regular refresher courses prior to engaging in any field effort. Certificates for each person should be incorporated into the site HASP. Additionally, all field personnel should have medical clearance in accordance with the HASP.

5.0 Equipment and Supplies

5.1 The equipment necessary for decontamination in the field may vary depending on the activities being conducted. A general list of equipment that may be utilized is as follows:

- Nitrile gloves
- Alconox (or other non-phosphate soap solution)
- Potable or distilled water
- 10% nitric acid rinse (only if sample is to be analyzed for metals)
- Hexane
- Isopropyl alcohol
- Paper towels
- Plastic (polyethylene) sheeting
- Containers for storage of decontamination liquids (e.g., poly-tank or 55-gallong drums)
- Approved Work Plan

6.0 Decontamination Activities

6.1 The following are the steps to be considered for decontamination of equipment and personnel during field investigation activities. The effectiveness of the decontamination process should be evaluated as part of the Work Plan.

7.0 Personnel Decontamination

7.1 The following steps should be followed for personnel decontamination:

- Remove any gross debris from gloves and place it in the designated waste accumulation point.
- Remove nitrile gloves, taking care not to contact the outside of the gloves, and place the gloves in the designated waste accumulation point.

8.0 Decontamination of Drilling Equipment

8.1 Decontamination of drilling equipment (e.g., augers, rods) should be conducted prior to and between drilling locations. This should be conducted in a manner to contain all fluids and cuttings, and may include a temporary decontamination pad specifically constructed for this purpose. Potable water should be available for the decontamination pad area. The following steps should be considered during the decontamination process:

- Position the equipment on the pad to avoid release of debris or overspray beyond the pad area.
- Don nitrile gloves and safety glasses.
- Remove gross debris from equipment and contain at a designated waste accumulation point.
- Thoroughly wash the equipment using a steam cleaner and potable water.
- Contain wastewater at a designated accumulation point.

Additional Steps for Non-Dedicated Sampling Equipment

- Don nitrile gloves
- Remove any gross debris or expendables and place it into the designated waste accumulation point
- Wash the equipment in a non-phosphate soap solution.
- Thoroughly rinse the equipment with potable or distilled water.
- Contain wastewater at a designated accumulation point.

9.0 Decontamination of Field Monitoring Equipment

- Don nitrile gloves.
- Remove any gross debris and place it into the designated waste accumulation point.
- Wipe the outside of the equipment with a moist towel.

10.0 Decontamination of Pumps and Electrical Equipment

10.1 Equipment involving internal components sensitive to decontamination fluids or electrical equipment, such as well pumps and water level indicators, that may be damaged by standard decontamination procedures can be decontaminated as follows:

- Place the submersible pump into a non-phosphate soap solution and operate the pump to ensure adequate rinsing of the internal pump assembly. For water level measurement devices, unreel the tape into the soap solution and agitate.
- Place the equipment into a potable water rinse. Operate pumps as described above to remove any residual soap solution.
- Rinse measurement tapes by agitating in potable or distilled water.

11.0 Investigation Derived Waste Management

IDW from the investigation activities should be properly managed to ensure safety to site personnel and to reduce the potential of impact to other areas of the site by the wastes. Wastes may include expendable sampling items such as gloves, plastic sheeting, paper towels, pump tubing, or bailers; media solids including soil cuttings or decontamination debris; or liquids such as well purge fluids or decontamination fluids. Should media be encountered that potentially meets the classification as a hazardous waste, these materials should be properly contained, labeled and stored until a formal waste characterization may be achieved. Final disposition should be based on the classification of the waste. Personnel should segregate all IDW according to the classifications identified above for final disposition. The following procedures should be considered to ensure proper management of IDW:

11.1 Expendable Materials

Expendable items are commercially acquired materials used in support of field activities. These materials may include but are not limited to packaging, paper towels, plastic sheeting, etc.

These materials should be placed into plastic garbage bags placed within the areas of activity or carried on the vehicle. Upon completion of the activity or when the bag has filled, the wastes should be placed into a designated disposal area for disposal of solid waste.

11.2 Solid Media Waste

Sampling-derived waste included in this category may include the following:

- Soil cuttings
- Solids accumulated during decontamination
- Personal protection equipment (PPE)

Unless otherwise authorized, cuttings should be placed into 55-gallon drums, sealed, labeled with the date, contents, and location; and subsequently transferred to a designated soil staging location until the waste can be adequately characterized and properly disposed.

Solids accumulated during decontamination should be placed into 55-gallon drums. Once filled, each drum should be sealed, identified with the

contents and date, and transferred to the onsite staging area for subsequent testing prior to disposal.

PPE should also be placed into a 55-gallon drum, sealed, labeled with contents and date, and transferred to the onsite staging area for subsequent testing prior to disposal.

11.3 Liquid Media Waste

Liquid wastes potentially generated during investigation activities may include the following:

- Drilling fluids
- Purged well water
- Decontamination fluids

Unless otherwise authorized, liquid wastes generated during the investigation should be containerized in 55-gallon drums, or other appropriate storage (i.e. polyethylene tanks). Containerized liquids should be labeled with the date, contents and location, and transferred to the staging pad for subsequent testing prior to disposal.

12.0 Data and Records Management

12.1 All data and information (e.g., location of decontamination pad, water source, site conditions) should be documented within site logbooks or field data sheets.

GROUNDWATER SAMPLING
STANDARD OPERATING PROCEDURE D.2

1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) is generally applicable to the collection of representative groundwater samples from permanent groundwater monitoring wells for laboratory analytical testing.

2.0 Summary of Method

2.1 The procedures presented herein address the collection of groundwater from permanent monitoring wells and include the following purging and sampling techniques:

- Low-flow sampling method
- Tap method

3.0 Health and Safety Issues

3.1 As with any activities associated with potential contaminants, work tasks should be conducted in strict accordance with Environmental Protection Agency (EPA), Occupation Safety & Health Administration (OSHA), client, and WESTON safety policy and procedure. This should include preparation of a site-specific Health and Safety Plan (HASP) to ensure that all aspects of potential risk are evaluated and properly addressed. A HASP has been prepared for the Study Area (see Appendix A of the Work Plan).

4.0 Personnel Qualifications

4.1 All field personnel with potential for exposure to contaminated media on site are required to take the 40-hour Health and Safety Training and regular refresher courses prior to engaging in any field effort. Certificates for each person should be incorporated into the site HASP. Additionally, all field personnel should have medical clearance in accordance with the HASP.

5.0 Equipment and Supplies

5.1 Equipment needed for collection of groundwater samples may include:

- Electronic water level indicator
- Logbook and waterproof pen

- Calculator
- Field purge forms and well location maps
- Safety equipment (e.g. safety shoes, safety glasses, hard hat, nitrile gloves, leather gloves, first aid kit)
- Decontamination equipment and reagents
- Groundwater quality monitoring equipment (e.g., pH, temperature, specific conductivity, turbidity, etc.)
- Wastewater holding tank or drums (if necessary)
- Tubing, clamps, couplings
- Grundfos Pump (2-inch or 4-inch Rediflo)
- Appropriate sample bottles and preservatives
- Chain-of-custody forms
- Coolers
- Plastic (polyethylene) sheeting
- Commercial plastic zip-sealed bags
- Sample bottle labels
- Approved Work Plan
- Disposable Teflon or polyethylene bailers
- Electrical cord
- Tool box with general tools (e.g., pliers, screwdrivers, wrenches)
- Gasoline-powered generator

6.0 Well Sampling Procedures

The general procedures to be applied for the sampling of monitoring wells may include the following general tasks:

- Well Preparation
- Well Purging
- Well Sampling

The following presents the procedures associated with each identified task.

6.1 Well Preparation

The following task should be conducted in preparation for well purging and sampling.

- Locate and confirm the identification of the well to be sampled.
- Locate the appropriate field purge form for the well.

- Organize equipment in the immediate area of the well.
- Inspect the condition of the well casing, lock and pad. Record the observations on the field purge form and/or the field logbook (as needed).
- Don nitrile gloves.
- Unlock the well casing and remove the riser plug to ensure the well conditions are stable.
- Use the electronic water level indicator and record the depth to groundwater (DTW) from an established reference point on the top-of-casing. If a reference point is not indicated, measurements should be recorded from the northern portion of the riser. Record the DTW information on the field purge form and/or field logbook.
- If the total depth of the well has not been established, use the water level indicator to measure the total depth of the well with reference to the top-of-casing. Record the information on the field purge form and/or field logbook.
- Decontaminate the water level indicator following the procedures outlined in the Decontamination SOP.

6.2 Well Purging Activities

6.2.1 Well purging will be performed using the low-flow sampling. The following procedure should be applied for low flow well purging:

- Don nitrile gloves.
- Select an adequate length of polyethylene tubing to reach the base of the monitoring well.
- Attach the tubing to the pump and carefully lower the pump and tubing into the well.
- Place the pump at the approximate midpoint of the screened or open borehole section of the well (unless fracture information is available in which case the pump should be placed across the noted fracture depth).
- Assemble the flow-through chamber for groundwater quality monitoring.
- Place the tubing into the top of the tank to collect the purged fluids (where required).
- Place the generator in a safe location downwind of the monitoring well and connect the pump to the pump regulator.

- Turn on the pump at a low rate (generally 100 to 300 milliliter/min [mL/min]).
- Record the start time on the field purge log and collect initial screening data for the parameters being monitored. Record the data on the field purge form.
- Monitor the drawdown of the water level. If the pump rate exceeds the recovery rate of the well, the pump rate should be lowered, as needed, to accommodate drawdown.
- If the drawdown is more than 0.3 feet and will not stabilize, the flow rate can be increased slightly until the well is pumped dry.
- Continue well purging and recording parameter screening data, collecting readings every three to five minutes.
- Stabilization should be considered achieved when three successive readings are within 0.2 units for pH, 3% for conductivity, 20 mv for oxidation reduction potential (ORP), 10% or 0.2 mg/l for dissolved oxygen, and 10% or 1.0 NTU for turbidity.
- Samples may be collected if the readings do not stabilize after five (5) well volumes are purged.
- For monitor wells that are purged dry, sampling activities should commence within 24 hours or when the water level has recovered within 80% of static conditions, whichever occurs first.

6.3 Well Sampling Activities

Sampling is the process of obtaining, containerizing, and preserving the groundwater sample after the purging process is complete. The precautions to be applied are as follows:

- Prior to sampling wells, personnel should thoroughly wash per the decontamination procedures outlined in the Decontamination SOP.
- Gloves should be changed prior to sample collection.
- Where possible, sampling materials and equipment should be disposable (or dedicated to a location) to avoid potential cross-contamination between wells.

6.3.1 Low Flow Sampling

The low flow sampling procedure includes:

- Prepare the sample containers and complete the labels.
- Don nitrile gloves.
- Disconnect flow-through cell used during purging to allow sample collection directly from tubing, decreasing flow rate as needed.
- Fill the bottles at a rate not to exceed the stabilized flow rate determined during purging until a sufficient volume is obtained to fill all sample bottles.
- During collection of VOC samples (if required by the Work Plan), the sample container should be filled by allowing the discharge water to flow down inside the container with minimal turbulence.
- Record the sample time on the field purge log and/or field logbook.

6.3.2 Tap Sampling

Tap sampling may be performed as needed as follows:

- Prepare the sample containers and complete the labels.
- Prior to sampling, the tap should be flushed for a minimum of five minutes.
- Don nitrile gloves.
- If flow control is adequate, collect samples directly into each sample bottle until sufficient volume is obtained to fill all sample bottles.
- Where possible, sampling should be conducted at the closest point to the well head and prior to any treatment.
- Record the sample time on the field purge log and/or field logbook.
- Parameter screening data at the time of sampling should be recorded on the field purge log and/or field logbook.

7.0 Sample Handling

7.1 Once the samples have been collected:

- Seal the containers, inspect the labels and place sample containers into cooler(s).
- Record all pertinent data in a site logbook or on a field data sheet.
- Complete the chain-of-custody form.
- Disconnect the equipment and remove the pump from the well (if applicable).
- Discard the expendable materials (e.g., tubing).
- Decontaminate non-disposable equipment via the procedures outlined in the Decontamination SOP.
- Secure the well and inspect the grounds for trash or loose equipment

8.0 Data and Records Management

All data and information (e.g., sample collection method used) must be documented on field data sheets or within site logbooks with permanent ink.

SOIL SAMPLING
STANDARD OPERATING PROCEDURE D.3

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is generally applicable to the development and application of a soil sampling program including discussion of methodology and equipment. The procedures discussed herein focus on the collection of surface soil samples (within approximately two feet from ground surface) utilizing manual hand-operated equipment and the collection of subsurface soil samples utilizing Geoprobe® and/or hollow-stem auger drilling techniques.

2.0 Summary of Method

2.1 This document has been prepared to assist personnel with the performance of specific tasks and procedures related to the collection of surface soil samples (0 to 2 feet below ground surface [ft bgs]) and subsurface soil samples (greater than 2 ft bgs). Where possible, Geoprobe drilling technology should be considered for subsurface soil sampling to minimize the quantity of investigative derived waste (IDW) generated during sampling activities. A hollow-stem auger drill rig should be utilized to install borings in locations where a Geoprobe cannot penetrate to the desired depth.

3.0 Health and Safety Issues

3.1 As with any activities associated with potential contaminants, work tasks should be conducted in accordance with applicable Environmental Protection Agency (EPA), Occupation Safety & Health Administration (OSHA), client and WESTON safety policy and procedures. This should include preparation of a site-specific Health and Safety Plan (HASP) to ensure that all aspects of potential risk are evaluated and properly addressed. A HASP has been prepared for the Study Area (see Appendix A of the Work Plan).

4.0 Personnel Qualifications

4.1 All field personnel with potential for exposure to contaminated media on site are required to take the 40-hour Health and Safety Training and regular refresher courses prior to engaging in any field effort. Certificates for each person should be incorporated into the site HASP. Additionally,

all field personnel should have medical clearance in accordance with the HASP.

5.0 Equipment and Supplies

5.1 To the extent possible, equipment used for sampling should be constructed of inert materials such as stainless steel or polyethylene. Ancillary equipment such as auger flights may be constructed of other materials.

5.2 Selection of equipment is usually based on the depth of the samples to be collected, but it is also controlled to a certain extent by the characteristics of the material. Equipment and supplies that may be required as part of this SOP include the following:

- Stainless steel hand-operated bucket auger
- Stainless steel or polyethylene scoops
- Stainless steel bowls or disposable plastic/polyethylene trays
- Stainless steel split-barrel sampler
- Plastic zip-sealed bags
- Survey stakes or survey flags
- Permanent markers
- Field logbook/field sheets
- Photoionization detector (PID)
- Area maps, ruler, waterproof pens
- Measuring tape (100 foot)
- Munsell Soil Color Reference Guide
- Shovel or post-hole diggers
- Safety equipment (e.g. safety shoes, safety glasses, hard hat, nitrile gloves, leather gloves, first aid kit)
- Plastic (polyethylene) sheeting
- Sample bottles, and labels
- Trip blanks
- Chain-of-custody forms
- Coolers
- Approved Work Plan
- Radio or cell phone
- Truck or suitable off-road vehicle

6.0 Sample Collection – Preparation

Pre-sampling preparation activities may include:

- Determine the extent of the sampling effort, the sampling methods to be employed, minimum sample volume requirements, and which equipment and supplies are needed.
- Obtain necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment (see decontamination SOP), and ensure that equipment is in working order.
- Use stakes or flags to identify and mark sampling locations. If required, the proposed locations may be adjusted based on site access, utility clearance and surface obstructions.

7.0 Sample Collection – Secondary Parameters

- Soil characterization data should be collected during soil sampling. Visual observations of soil color and texture, descriptions of soil horizons, moisture, the presence of any non-native material should be recorded on field data sheets or in the field logbook (as necessary).

8.0 Sampling Methodology

8.1 Surface Sampling Procedures

1. This discussion of soil sampling methodology is generally applicable to the collection of surface soil samples using scoops or hand augers.
2. Sampling locations may be tentatively located prior to mobilization to the site based on historic records, aerial photographs, and site drawings. Upon entering the field, the proposed area should be evaluated to confirm that samples collected from the area meet the objectives of the investigation in accordance with the Work Plan. The following procedures may be applied to the site for sampling:
    - Conduct reconnaissance of the area to locate appropriate sample locations (modify locations if necessary as previously discussed).
    - Designate the location with a unique sample identifier and place a stake or survey flag at the location with the sample site identification.

- Don gloves and prepare equipment. If hand augers are to be used, leather gloves are permitted provided there is no contact with the sampled media.
- Begin construction of the sample boring by removing the soil horizon (upper soil horizon containing the vegetative root mat generally high in organic debris).
- Continue the boring until the desired depth is achieved.
- Collect soil from the sampling interval using decontaminated or disposable equipment (scoop or auger).
- Collect grab samples (as required in accordance with the Work Plan).
- For composite sampling (excluding VOCs), place the soil from the sampling into a decontaminated disposable tray for blending (blend the soil until the soil is adequately homogenized).
- Adequately describe the sample including sample depth, soil color, texture, moisture content and a soil description.
- When adequate volume is achieved, blend the soil in the bowl until the soil is adequately homogenized.
- Place the soil media into appropriately prepared laboratory containers.
- Seal, label, and place the containers into a cooler.
- Adequately describe the sample location. May include site setting, vegetation, drainage conditions, depth to sampling location, and a soil description.
- Complete the chain-of-custody.
- Decontaminate the sampling equipment (according to the procedures outlined in to the Decontamination SOP).
- Dispose of expendable items in the waste allocation area and backfill sampling site (as necessary).

8.2 Subsurface Soil Sampling

This discussion of soil sampling methodology is applicable to the collection of subsurface soil samples using Geoprobe or hollow-stem auger drilling techniques using stainless steel split-barrel samplers. The following procedures may be applied to the site for sampling:

- Don gloves and expose the surface soil by either pulling barrels apart or cutting the boring liner.
- Follow necessary Work Plan procedures for logging of the soil core and sample collection.
- Adequately describe the sample including sample depth, soil color, texture, moisture content and a soil description.

- Collect grab samples (as required in accordance with the Work Plan).
- For composite sampling (excluding VOCs), place the soil from the sampling into a decontaminated disposable tray for blending (blend the soil until the soil is adequately homogenized).
- Place the soil media into appropriately prepared laboratory containers.
- Seal, label, and place the containers into a cooler.
- Complete the chain-of-custody.
- Decontaminate the sampling equipment (according to the procedures outlined in to the Decontamination SOP.

9.0 Data and Records Management

All data and information (e.g., sample collection method used) must be documented on field data sheets or within site logbooks.

CONSTRUCTION OF MONITORING WELLS
STANDARD OPERATING PROCEDURE D.4

1.0 Scope and Application

1.1 This standard operation procedure (SOP) is generally applicable to the development and application of procedures related to the installation of permanent groundwater monitoring wells.

2.0 Summary of Method

2.1 This document has been prepared to assist personnel with the performance of specific tasks and procedures related to the installation of permanent groundwater monitoring wells via the hollow-stem auger drilling technique.

3.0 Health and Safety Issues

As with any activities dealing with potential contaminants, work tasks should be conducted in accordance with applicable Environmental Protection Agency (EPA), Occupation Safety & Health Administration (OSHA), Client, and WESTON safety policy and procedures. This should include preparation of a site-specific Health and Safety Plan (HASP) to ensure that aspects of potential risk are evaluated and properly addressed. A HASP has been prepared for the Study Area (see Appendix A of the Work Plan).

4.0 Personnel Qualifications

4.1 All field personnel with potential for exposure to contaminated media on site are required to take the 40-hour Health and Safety Training and regular refresher courses prior to engaging in any field effort as well as other training as determined in the site-specific HASP. Certificates for each person should be incorporated into the site HASP. Additionally, all field personnel should have medical clearance in accordance with the HASP.

5.0 Equipment and Supplies

5.1 The equipment necessary in the field will vary depending on the activities being conducted. A general list of equipment that may be utilized is as follows:

- Intrusive equipment used to install monitoring wells including drilling tools, augers, rods, etc.
- Plastic sheeting
- Survey stakes or survey flags
- Permanent markers
- Field logbook/field sheets
- Area maps, ruler, waterproof pens
- Safety equipment (e.g., safety shoes, safety glasses, hard hat, nitrile gloves, leather gloves and first aid kit)
- Plastic (polyethylene) sheeting
- Drums or other containers for investigative derived waste
- Approved Work Plan
- Radio or cell phone
- Truck or suitable off-road vehicle

6.0 Decontamination of Drilling Equipment

6.1 Decontamination of drilling equipment (e.g., augers, rods, etc.) should be conducted prior to and between drilling locations to prevent any cross contamination. Fluids and soil derived during the decontamination activities should be containerized. This may include a temporary decontamination pad specifically constructed for this purpose. The following steps should be considered during the decontamination process:

- Position the equipment on the pad to avoid release of debris or overspray to adjacent areas.
- Don nitrile gloves and safety glasses.
- Remove gross debris from equipment and contain at a designated waste accumulation point.
- Thoroughly wash the equipment using steam cleaner and potable water.
- Contain all wastewater at a designated accumulation point.

7.0 Well Construction

7.1 Once the borehole is constructed to the desired depth, the well material should be inserted as soon as possible to prevent potential blockage within the borehole due to collapse or native material infiltration. The following procedures should be considered for well installations:

- Well materials should be new in factory sealed material to prevent potential contamination from outside sources prior to installation.
- Personnel handling the materials should wear adequate personal protective equipment (e.g., gloves) to prevent contact with the material and the potential introduction of contaminants.
- Screens should be selected with appropriate length and slot diameter to allow adequate recharge and to prevent excessive native material infiltration into the well.

7.2 Once the well components are in place the well will be constructed in accordance with state regulations. The well construction will consist of clean quartz sand constructed across the screened interval to serve as a filter pack between the screen and adjacent native material.

- Sand may be placed as either dry or as slurry into the annulus using a tremie pipe. Potable water may be added to facilitate sand placement. The source of the water should be documented.
- Wells may be constructed within augers to prevent collapse of the annulus.
- The augers may be gradually removed as construction of the annulus continues until a depth of approximately 20 feet below grade. At that point, depending on the competency of the soil, the augers may be completely removed for final annulus construction.
- For borings of less than 20 feet (depending on soil conditions), well construction may be conducted without use of the augers or tremie pipe. The well material may be placed into the open borehole, followed by placement of the filter sand. The sand may be placed directly into the top of the borehole and allowed to settle by gravity. The depth to sand should be measured to ensure that bridging does not occur and to document final material depth.

7.3 Following sand filter pack placement to a minimum of one-foot above the top of the screen, a low-permeability seal (e.g., bentonite slurry, choke sand) should be placed on top of the filter pack. As with the sand pack,

seal should be placed by way of the tremie pipe method to minimize the risk of bridging during construction.

- The seal should be a minimum of 2 feet thick.
- In shallow borings, less than approximately 20 feet and depending on soil conditions, the slurry may be introduced by slowly pouring the seal into the top of the borehole.
- Pellets, if used in shallow wells, should be allowed to hydrate a minimum of 30 minutes prior to the introduction of the final grout seal. A small amount of potable water may be added to accelerate the hydration of the pellets (especially if zone pellets are added is above the water table).

7.4 The remaining annular space between the seal and ground surface should be filled with a cement/bentonite grout mixture.

- The grout should be introduced as a slurry using a tremie pipe and pressure pumping, introducing the grout at the base of the annular space.
- The annulus should be filled to approximately two-feet below grade to allow placement of the protective casing and/or concrete pad.

7.5 Following placement of the aforementioned materials into the annular space, the well should be completed in such a way so as to protect and maintain the integrity of the well.

- Flush-mounted wells should be constructed with the well riser at or slightly below ground level.
- Above-ground wells should be completed with approximately 2 feet of riser extending above grade.
- A protective, lockable casing should be placed over the well riser and into a concrete pad staged away from the well.
- In areas of high vehicle traffic or areas of limited view, steel posts or bollards may be installed around the well pad to prevent damage to the well.

8.0 Well Development

8.1 After each new monitoring well is installed, it should be developed (using a submersible pump or similar technique) to surge and pump the well until sediment production is negligible.

- New monitoring wells should be allowed to set for at least 24 hours prior to development.

9.0 Investigation Derived Waste Management

Investigation derived wastes (IDW) generated during the investigation activities should be properly managed to ensure safety to site personnel and to reduce the potential of impact to other areas of the site by the wastes. Wastes may include expendable sampling items such as gloves, plastic sheeting, paper towels; media solids including soil cuttings, decontamination debris, or sediment residuals; or liquids such as well purge fluids or decontamination fluids. Should media be encountered that potentially meets the classification as a hazardous waste, these materials should be properly contained, labeled and stored until a formal waste characterization may be achieved. Final disposition should be based on the classification of the waste. Personnel should segregate all IDW according to the classifications identified above for final disposition. The following procedures should be considered to ensure proper management of IDW:

9.1 Expendable Materials

Expendable items are commercially acquired materials used in support of field activities. These materials may include but are not limited to well material packaging, paper towels, plastic sheeting, etc.

These materials should be placed into plastic garbage bags placed within the areas of activity or carried on the vehicle. Upon completion of the activity or when the bag has filled, the wastes should be placed into a designated disposal area(s).

9.2 Solid Media Waste

Sampling-derived waste included in this category may include the following:

- Soil cuttings
- Solids accumulated during decontamination
- Personal protection equipment (PPE)

Unless otherwise authorized, cuttings should be placed into 55-gallon drums, sealed, labeled with the date, contents, and location; and subsequently transferred to a designated soil staging location until the waste can be adequately characterized and properly disposed.

Solids accumulated during decontamination should be placed into 55-gallon drums. Once filled, each drum should be sealed, identified with the contents and date, and transferred to the onsite staging area for subsequent testing prior to disposal.

PPE should also be placed into a 55-gallon drum, sealed, labeled with contents and date, and transferred to the onsite staging area for subsequent testing prior to disposal.

9.3 Liquid Media Waste

Liquid wastes potentially generated during investigation activities may include the following:

- Drilling fluids
- Purged well water
- Decontamination fluids

Unless otherwise authorized, liquid wastes generated during the investigation should be containerized in 55-gallon drums, or other appropriate storage containers (i.e. polyethylene tanks). Containerized liquids should be labeled with the date, contents and location, and transferred to the staging pad for subsequent testing prior to disposal.

10.0 Data and Records Management

10.1 All data and information (e.g., location of decontamination pad, water source, site conditions) should be documented within site logbooks or field data sheets.