NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

Division of Environmental Remediation, Remedial Bureau E
625 Broadway, 12th Floor, Albany, NY 12233-7017
P: (518) 402-9813 I F: (518) 402-9819
www.dec.ny.gov

November 17, 2020

Mr. Greg Haack, PE
Sr. Facilities Engineer
Capital Projects and Facilities Engineering
Corning Incorporated
Corning, New York 14831

Subject: Site No. 851046 City of Corning Study Area Operable Unit 4 Flood Control Areas Site Characterization Work Plan

Dear Mr. Haack,

The New York State Department of Environmental Conservation (the Department) in consultation with the New York State Department of Health (NYSDOH) have reviewed the Site Characterization Work Plan dated November 12, 2020 for Study Area Operable Unit 4 Flood Control Areas, Site No. 851046 and find it acceptable.

Please continue to provide the Department with written schedule updates as well as provide the Department a minimum of two weeks' notice prior to any mobilization to the Study Area OU4 Flood Control Areas.

Sincerely,

Samantha Salotto
Assistant Engineer (Environmental)
Remedial Bureau E, Section A
Division of Environmental Remediation

Ec: M. Cruden
D. Harrington
M.J. Crance, DFW
D. Loew, OGC
D. Pratt, Region 8
K. Cloyd, Region 8
J. Robinson, NYSDOH

M. Doroski, NYSDOH
J. Deming, NYSDOH
M. Vetter, Parsons
J. Novotny, Corning Incorporated
P. Zimmerman, Integral for Corning Incorporated
M. Greenblatt, Integral for Corning Incorporated
J. Marsh, Integral for Corning Incorporated

CHARACTERIZATION WORK PLAN:
STUDY AREA OPERABLE UNIT 4 FLOOD CONTROL AREAS

NYSDEC Project ID 851046, Corning, New York

Prepared for
Corning Incorporated
Corning, New York

Prepared by

1001 6th Avenue
11th Floor
New York, NY 10018

November 12, 2020

Affiliated with Integral Consulting Inc.

CERTIFICATION

I, Marcia Greenblatt, Ph.D., P.E., certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375 and that this Characterization Work Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10).

Signature

November 16, 2020

Date

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

amsl	above mean sea level
AOC	area of concern
bgs	below ground surface
CAMP	Community Air Monitoring Plan
cfs	cubic feet per second
Corning	Corning Incorporated
CSM	conceptual site model
DGPS	digital global positioning system
DUSR	data usability summary report
EDD	electronic data deliverable
FEMA	Federal Emergency Management Agency
FFS/AA	Focused Feasibility Study / Alternatives Analysis
FOIL	Freedom of Information Law
GPS	global positioning system
HASP	Health and Safety Plan
IDW	investigation-derived waste
Integral	Integral Engineering, P.C.
ISMP	Interim Site Management Plan
NWI	National Wetlands Inventory
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
ORP	oxidation-reduction potential
OU4 Work Plan	Characterization Work Plan: Study Area Operable Unit 4 Flood Control Area
OU	operable unit
PFAS	per- and polyfluoroalkyl substances
PID	photoionization detector
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance and quality control

RAWP	Remedial Design/Remedial Action Work Plan
SCO	soil cleanup objective
SMP	Site Management Plan
SOP	standard operating procedure
SVOC	semivolatile organic compound
TAL	target analyte list
TestAmerica	TestAmerica Laboratories, Inc. in Buffalo, New York
TOC	total organic carbon
USACE	U.S. Army Corps of Engineers
USCS	Unified Soil Classification System
USFWS	U.S. Fish and Wildlife Service
USGS	U.S. Geological Survey
Weston	Weston Solutions, Inc.

1 INTRODUCTION

Corning Incorporated (Corning) entered into an Order on Consent and Administrative Settlement: CO 8-20171204-140, December 4, 2017 (the Remedial Order) with the New York State Department of Environmental Conservation (NYSDEC) to perform characterization and remedial activities within the Corning Study Area. This Characterization Work Plan: Study Area Operable Unit 4 Flood Control Areas (OU4 Work Plan) has been prepared by Integral Engineering, P.C. (Integral) with support from Weston Solutions, Inc. (Weston) on behalf of Corning to plan and perform initial characterization activities in Operable Unit 4 (OU4), pursuant to Section II.C of the Remedial Order.

The Study Area is located in the City of Corning, New York, as illustrated on Figure 1-1. The Study Area consists of 201 acres bounded by the Chemung River to the south; Post Creek and Interstate 86 to the east and north; and Center Way, the Guthrie Medical Center, and the City of Corning Fire Department to the west. The Remedial Order divides the Study Area into five operable units (OUs): the Residential Area (OU1), the Residential Area at the Eastern End of Corning Boulevard (OU2), the School/Community Use Areas (OU3), the Flood Control Areas (OU4), and the Residential Expansion Area (OU5). The Study Area OUs are depicted on Figure 1-2.

OU4 consists of approximately 73 acres owned by the City of Corning. OU4 includes areas along the eastern and southern boundaries of the Study Area, consisting of the earthen berm and lands between the eastern boundary of OU1, OU2, OU3 and Post Creek and Highway 17/Interstate 86 to the east as well as the berm and lands between the southern boundary of OU3 and the Chemung River (Figure 1-3).

1.1 REGULATORY AND INVESTIGATORY BACKGROUND

Corning and NYSDEC activities conducted to date for the Study Area have involved OUs 1, 2, 3, and 5 and include the following:

- Study Area—Corning submitted the Study Area Characterization Work Plan (Weston 2014a) on June 27, 2014. NYSDEC approved of the Study Area Characterization Work Plan as indicated in Order B8 XXXXX. NYSDEC-approved addenda to the Study Area Characterization Work Plan for additional field investigation activities in OU1, OU2, and OU3 were issued in 2014, 2015, and 2017 (Weston 2014b, 2015a,b, 2017a).
- OU1, OU2, and OU5—On July 12, 2017, NYSDEC issued a Final Decision Document (NYSDEC 2017a) outlining investigation and remediation activities for OU1, OU2, and OU5. Supporting documents include the January 9, 2018, Study Area Pre-Design Investigation Work Plan; the March 23, 2017, Focused Feasibility Study / Alternatives

Analysis (FFS/AA; Weston 2017b); and the April 6, 2018, Remedial Design/Remedial Action Work Plan (RAWP; Weston 2018) that describes the methods for determining the extent of remediation and for implementing remediation within OU1, OU2, and OU5. Corning will also prepare an Interim Site Management Plan (ISMP) to guide activities until implementation of the RAWP has been completed, after which a Site Management Plan (SMP) will be developed and implemented.

- OU3—On July 11, 2017, NYSDEC issued a Final Decision Document (NYSDEC 2017b) outlining investigation and remediation activities for the school and community use areas (OU3). Interim remedial action work plans for each of the OU3 properties, Corning/Painted Post School District (Weston 2016a), Corning Memorial Stadium (Weston 2016b), and Corning Christian Academy (Weston 2016c), were approved by NYSDEC and completed in the summer of 2017.

The Final Decision Documents identified the contaminants of concern for the Study Area characterization activities as lead, cadmium, arsenic, and semivolatile organic compounds (SVOCs) (NYSDEC 2017a,b).

For OU4, the Remedial Order requires submittal and implementation of a characterization work plan, presented in this document, followed by submittal of a characterization report.

1.2 OU4 CHARACTERIZATION OBJECTIVES

The objectives of the OU4 characterization are consistent with those specified in the NYSDEC-approved Study Area Characterization Work Plan (Weston 2014a) and are summarized below:

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

These characterization activities were developed and approved by NYSDEC in compliance with NYSDEC guidance (DER-10; NYSDEC 2010) and are intended to determine if a threat to public health or the environment exists within OU4 and if further investigation is warranted. The steps in a characterization are a records search and a field characterization, followed by a decision of either no further action or additional investigation (NYSDEC 2010, Section 3.5). Characterization is intended to determine if the conditions identified in the investigation "…can be attributed to disposal in an area of concern (AOC) on the site identified by the site records

search and if the site is a contaminated site requiring further investigation and remediation" (NYSDEC 2010).

The OU4 characterization will be performed in a phased manner that is consistent with NYSDEC's Technical Guidance for Site Investigation and Remediation (DER-10; NYSDEC 2010) and was used as the overarching guidance for this OU4 Work Plan. As described in DER-10 (Section 3.1), characterization "is designed to determine whether a site poses little or no threat to public health or the environment" and "includes the gathering of sufficient information to determine whether the site requires further investigation or remediation."

Consistent with DER-10, the first phase of the OU4 characterization has been developed based on review of historical records, site-specific field observations, and regional studies. The first phase targets identified areas of potential disturbance to determine the presence of fill material; these are the areas that are most likely to have a pathway for exposure if fill material is present. The results of the first phase will inform the sampling design for the second phase.

In this work plan, "fill" is used to refer to material that contains ash, brick, and/or glass. A layer of fill material is defined as nonnative material containing ash, brick, and/or glass with a thickness of greater than 1 in.

1.3 WORK PLAN ORGANIZATION

This Work Plan follows the guidance for investigation work plans as described in DER-10 (Section 3.3). The Work Plan guides the field investigations and includes the following:

- Section 1—Introduction
- Section 2—Area Background
- Section 3—Environmental Setting
- Section 4—Conceptual Site Model
- Section 5—Characterization Activities
- Section 6—Project Management.

The following appendices are included in the Work Plan:

- Appendix A—Health and Safety Plan (HASP)
- Appendix B—Community Air Monitoring Plan (CAMP)
- Appendix C—Quality Assurance Project Plan (QAPP)
- Appendix D—Standard Operating Procedures (SOPs), including procedures for managing investigation-derived waste (IDW)

- Appendix E—Aerial Photographs
- Appendix F—Biological Survey Approach.

2 AREA BACKGROUND

Consistent with DER-10 (Section 3.1), historical records were reviewed to characterize past uses and activity in and around OU4. This history provides important context for interpreting site observations and a basis for developing the field sampling plan.

2.1 STUDY AREA HISTORY

The City of Corning has a long history of manufacturing, particularly in brick and glassmaking. Historical references indicate that, in the late 1800s and early 1900s, one of the country's largest brick manufacturers and more than 60 glass manufacturers were located in the City of Corning (Dimitroff and Janes 1991; Sinclair and Spillman 1997), including Corning Incorporated, which was formerly known as Corning Glass Works. During that time frame, coal was the primary fuel source in the Corning, New York, area, and most of the local industries and municipalities used coal to heat their furnaces. In the early 1900s, when natural gas was introduced to the region, some industries converted their fuel sources to natural gas.

Between 1949 and at least 1968, the City of Corning operated a municipal incinerator that created significant volumes of ash. Historical City Council meeting minutes indicate that the City applied ash and cinders to roadways within the City to control ice in the winter months during, at least, the mid-1950s (City of Corning 1936, 1941, 1950, 1958, 1959). These records also indicate that when land within the Study Area (now comprising OU3) was being considered for redevelopment as a school in the late 1950s, the City of Corning stated that it would require "a considerable amount of work and expense involved in filling and grading to render the track suitable for recreational and educational purposes." (City of Corning 1950). Ultimately, a school, which opened in 1962, was constructed on this portion of the Study Area.

2.1.1 Flooding History

The Chemung River, near the City of Corning, is prone to flooding due to its steep slopes and the tendency for flash flooding in its tributaries (NYSDEC 2007). Multiple major storm events have occurred in the region, dating back to the eighteenth century (Copp 1975), which resulted in major flooding of the Chemung River, and in some cases led to the destruction of flood control infrastructure. The "Finger Lakes Flood" of 1935 caused extreme damage and loss of life in the City of Corning and the region, leading the U.S. Army Corps of Engineers (USACE) to raise the level of the flood control berms in the early 1940s (Copp 1975). In June 1972, Tropical Storm Agnes led to overtopping of flood protection works, inundation of the Study Area and damage to the flood control berm in the southeast corner (EADS 1979; Copp 1975). In September 1975, Tropical Storm Eloise occurred (EADS 1979; Copp 1975). In June 1976, more than 5 in. of rain fell in 8 hours (EADS 1979; Copp 1975). Both storms resulted in flooding less

severe than that of Tropical Storm Agnes (EADS 1979). Rising subsurface water due to the high stage of the river caused the majority of flooding from Tropical Storm Eloise (EADS 1979). Problems with interior drainage caused flooding during the storm in June 1976 (EADS 1979).

2.1.2 Flood Control History

Due to recurring flooding, flood control structures line many of the rivers and tributaries in the region. Prior to 1901, a flood control berm was present along the Chemung River and Post Creek along the perimeter of the OU4 area (Rockwood 1901). By 1921, a retention wall existed between River Street and the Chemung River (EDR 2014). In 1937, the first federal levee was constructed in the City of Corning (EADS 1979, p. 7), and in 1942, federal protection works enlarged the levee along the Chemung River and Post Creek (USACE 1941, 1973). These improvements were designed to provide increased protection to the City of Corning and Painted Post against flooding. The levee was expanded in three units in the City of Corning consisting of 43,000 ft of earthen berms and 3,300 ft of concrete wall, as well as channel excavation and realignment, pumping station, and drainage structures along the Chemung and Cohocton rivers and Cutler, Post, and Gordon creeks (EADS 1979; USACE 1941). Such construction efforts required significant volumes of material from known and uncertain origin, the removal or relocation of material deemed unsuitable as foundation for earthworks, the creation and filling of borrow areas from which soils suitable for construction were obtained, as well as other potential grading and filling activities. Flood control construction activities are summarized in Figure 2-1.

Drainage controls along the levee were added prior to and during 1956 in the areas of OU4 and OU5 (USACE 1956). In 1973, the flood control berm was repaired due to damages incurred during Tropical Storm Agnes, and a sanitary siphon through the berm was replaced (USACE 1973; Dalton 1974).

2.2 HISTORICAL RECORDS REVIEW

Integral and Weston reviewed available historical records pertaining to OU4 to evaluate the potential for a layer of fill material containing ash, brick, and/or glass to be present within OU4. This included a review of available historical aerial photographs and a review of files provided by NYSDEC under a Freedom of Information Law (FOIL) request pertaining to the flood control structures (earthen flood control berms) constructed in the Study Area by USACE and maintained by NYSDEC. The results of the historical records review are used to identify locations to be sampled in the field investigation (see Section 5).

2.2.1 Aerial Photograph Review

To evaluate the potential for a layer of fill material containing ash, brick, and/or glass within OU4, Integral and Weston conducted a review of available historical aerial photographs for the Study Area. Historical aerial photos were reviewed with a specific focus on evidence of construction or disturbance activities that may have resulted in placement of fill. 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 change can be observed, which could indicate activities such as preparation for construction, grading, deposition, borrow, standing water, distressed vegetation, roads, trails, etc. (It is important to recognize the season when the photo was taken as lack of leaves on trees may not be due to activities but simply due to seasonal effects.)

Aerial photographs from 17 dates over the period of 1938 to 2014 were reviewed for OU4 (Appendix E). For brevity, photos for the following nine dates are discussed here and included on photo chronology figures (Figures 2-2 and 2-3):

May 11, 1942
April 16, 1952
July 11, 1955
September 7, 1962
October 8, 1964
March 30, 1968
May 7, 1973
September 22, 1991
March 21, 2012

The full set of aerial photographs was evaluated; photographs not discussed here are consistent with observations made below. The 1942 photo shows the earthen flood control berm in OU4 at the approximate location where it exists today, consistent with information that indicates flood control berms were built prior to 1901 (Rockwood 1901). A road crosses over the earthen flood control berm to OU4 southeast of the sports stadium (which is visible in all photos, although its primary use has evolved from baseball to football) and north of the western end of OU4. The road goes diagonally east–southeast after crossing the berm, but its course has evolved over the years from heading straight to the river prior to the 1973 photo, to moving directly west, and then fading in use since approximately 1991 (Figure 2-2, orange star).

The location of Post Creek has changed over time (Figure 2-3; the blue lines indicate the current creek path). In the 1942 aerial photograph, Post Creek appears to meander along the eastern

side of the flood control earthen flood control berm in the northern portion of OU4, and then flows to the east–southeast and parallels the hillside along the creek. In the 1952 aerial photograph, Post Creek appears to have been straightened along what looks to be a railroad east of the Study Area. In the 1991 aerial photograph, Post Creek appears to have been altered again and the channel rerouted west to allow for the construction of Highway I-86, consistent with other documents suggesting that the rerouting occurred in the early 1980s (USDOT 1988). The lower portion of Post Creek has remained stable over time as evidenced by the change from low-growing vegetation to larger plants that occurs to the southeast of the green stars on Figures 2-2 and 2-3. This vegetated area between Post Creek and the Chemung River is consistently observed in all the photos after 1955, and can be seen developing in the photos prior to 1955.

The aerial photos do show some modifications to the berms at various times, as shown on Figure 2-2. These include the 1952 photo, which shows the addition of material at the top of the berm along the Chemung River, and the addition of material along the top of the berm and modifications associated with berm construction along the foot of the berm along Post Creek. The 1955 photo also shows modifications along the bottom of the berm along Post Creek and an apparent road going over the berm at the eastern end of Corning Boulevard to access the floodplain area along Post Creek.

The 1973 aerial photo shows disturbed areas along the berm, which were likely associated with berm and sanitary siphon damage and repairs following floods from Tropical Storm Agnes in June 1972 (purple star on Figure 2-2, only on the 1973 photo), with indications of repairs at the southeast corner of OU3 on the Chemung Creek side. The 1973 photo shows additional roads, disturbed ground, and potentially a small catch basin near the easternmost portion of the Chemung River side of the berm, around the corner from the downstream end of Post Creek. Historical records (NYSDEC 1973) indicate that sections of the berm along the downstream end of Post Creek were washed out during Tropical Storm Agnes flooding and repaired in 1972 and 1973; this description may be related to the construction activities seen in the 1973 photo, or may refer to other activities not observed in the photos.

The aerial photo analysis shows that the floodplain has been largely stable over time, evidenced by the mature forested area along Post Creek and the unchanged location of the upper portion of the dirt road into OU4 in the southwest corner. Temporal changes for OU4 observed in the sequence of photos include three general disturbances since the 1930s: construction associated with the berms in the 1940s, rerouting of Post Creek in the 1950s and 1980s associated with road construction, and repairs in 1973 associated with the Tropical Storm Agnes floods. Other than these focused construction efforts, little evolution and disruption of the land in OU4 is evident from aerial photos and there is not a strong indication of areas of fill or disturbance within OU4. A summary of OU4 construction activities is presented in Figure 2-1.

2.2.2 NYSDEC Flood Control Files

Files pertaining to the earthen flood control berm in the Study Area provided by NYSDEC under a FOIL request were reviewed. According to the files, USACE identified areas in the floodplains along Post Creek and the Chemung River that could be utilized as borrow pits for the earthen flood control berm reconstruction project during the 1940s (USACE 1941). The areas identified include an area south of OU3 between the existing earthen flood control berm and the Chemung River's bank, and areas along both sides of Post Creek (USACE 1941).

According to exploration logs (which include test pits, borrow pits, core drill holes, and auger borings) for foundation test pits completed during the earthen flood control berm reconstruction project in the 1940s, one test pit (Foundation Test Pit Number 20) out of 19 foundation exploration logs (Figure 2-4) contained material at the surface only, described as "fill, cinders, glass, etc." Foundation Test Pit Number 20 was located along the southern earthen flood control berm, nearly due south of the intersection of Pyrex Street and Corning Boulevard, in an area that is now located on the City of Corning Memorial Stadium property. The logs in OU4 were completed over depths of 7 to 42 ft; 17 of the 19 in OU4 contained no fill and 1 contained fill material consisting of plain soil or wood (TP-18). Foundation logs for the locations in OU4 are presented on Figures 2-5a–c and summarized in Table 2-1.

In the 1940s, to confirm that subsurface soil was acceptable for the reconstruction of the earthen flood control berm and to tie the reconstructed earthen flood control berm to the existing subsurface soil, USACE excavated a 5-ft wide, 6-ft deep inspection trench along the existing earthen flood control berm. The new earthen flood control berm was then constructed over the inspection trench. Footnotes on USACE maps indicate that the "Inspection Trench [is] to be deepened or widened as directed if unsuitable material is encountered," suggesting this material was excavated and replaced with suitable fill (USACE 1941). In addition, the USACE inspection reports state that "where existing unsuitable fill and other material were found in the foundation, they were reportedly removed and replaced with impervious fill" (USACE 2011).

The USACE 1941 drawings also identified an area for "spoil" material within OU4 along Post Creek (Figure 2-4). Spoil material areas were located along the west bank of Post Creek. The composition of this spoil material is unknown; a note on the USACE 1941 drawings states that "All excess excavated fill shall be hauled to spoil areas"; however, no clear definition of the nature of the spoil material was provided in the USACE document (e.g., if the fill contained ash or cinder or other specific material).

The USACE 1941 drawings include borrow area cross sections, which identify areas evaluated to find material used for backfill (USACE 1941). According to general notes, excavated material was stockpiled for reuse as backfill, and excess material would be hauled to spoil areas. If undesirable fill existed in inspection trenches, the trenches would be excavated deeper and wider (USACE 1941); this suggests that, if undesirable fill materials were encountered, they

were removed and replaced with suitable backfill. For the Chemung bank backfill, the material used to enlarge the berm came from an area upstream of the Study Area, west of the railroad bridge (USACE 1941).

2.3 OU4 FIELD RECONNAISSANCE

Field reconnaissance and visual inspection of OU4 were performed by Weston on September 8 through 10, 2014, and by Integral and Weston on May 31, 2018. Observations made during both field reconnaissance/visual inspections confirmed that the floodplain along the northern bank of the Chemung River slopes gently from the base of the earthen flood control berm to the river edge along the western, upstream portion of OU4. Further downstream toward the confluence of Post Creek in the southeastern corner of OU4, the Chemung River channel is more deeply incised with the low-flow water level approximately 6 to 8 ft below the surrounding floodplain. Stormwater conveyances through the earthen flood control berm were observed, appearing to facilitate flow of stormwater drainage from neighboring residential and school areas to the river and Post Creek; flood control gates to prevent backflow of river and creek water during flood events were observed on the outfalls.

During the field reconnaissance of OU4 in 2018, overall, thick vegetation with grasses were observed over much of the area along Chemung River (Figure 2-6, bottom and left photographs). In the eastern portions of OU4, herbaceous and woody vegetation was present, especially along Post Creek (Figure 2-6, top right photograph). Along the channels of both Post Creek and the Chemung River, patches of bare, unvegetated shoreline are present at non-flood conditions (Figure 2-7, top and bottom photographs, respectively). Wetland areas are present along the fringe of both Post Creek and the Chemung River. During the 2014 field reconnaissance, maintained and mowed turf grass was observed over much of OU4, with herbaceous and woody vegetation, similar to the 2018 observations. No fill material containing ash, brick, and/or glass was observed during the field reconnaissance within the areas observed.

Additional information about features observed during field reconnaissance is provided in the description of the environmental setting in Section 3.

2.4 STUDY AREA CHARACTERIZATION

In 2012, during a capital improvement project at the Corning-Painted Post High School located in OU3, within the southern portion of the Study Area, fill material that the Corning-Painted Post School District described as containing ash, brick, and glass was encountered in the subsurface soil. During the capital improvement project, the Corning-Painted Post School District's consultant tested excavated material to determine appropriate disposal methods. A review of a summary of the analytical results for these samples of excavated material, prepared by the Corning-Painted Post School District's consultant for NYSDEC, indicates more than

200 samples were collected and analyzed for various chemical constituents. The primary constituents that exceeded the soil cleanup objectives (SCOs) in the excavated materials are lead, cadmium, and arsenic. Some of the excavated material was classified as hazardous waste based on levels of lead and cadmium; this material was removed and disposed.

2.4.1 Characterization Activities

Commencing in 2014, Weston performed characterization activities in the Study Area under an Order on Consent and Administrative Settlement between Corning and NYSDEC, dated June 27, 2014 (June 2014 Order on Consent). Characterization activities were performed in accordance with the Study Area Characterization Work Plan dated June 2014 (Weston 2014a), and three addenda (Weston 2014b, 2015a,b). Collectively, the June 2014 Study Area Characterization Work Plan and its addenda, as modified, amended and approved by NYSDEC, are referenced herein as the Study Area Work Plan. Characterization activities were also performed by NYSDEC in OU1 and OU2 in 2014 and 2015. In 2015, Arcadis CE, Inc., on behalf of NYSDEC performed characterization activities in OU5. The objectives of the Study Area characterization activities were to assess the nature and extent of layers of fill material containing ash, brick, and/or glass that were encountered within the Study Area, and to collect data necessary for understanding current conditions and associated potential exposure pathways.

Characterization activities in OU1, OU2, OU3, and OU5 of the Study Area are substantially complete. Validated analytical data for samples collected by Weston are submitted to NYSDEC in data usability summary reports (DUSRs) as completed throughout the characterization program. NYSDEC has provided data to Weston for its characterization samples collected in the Study Area.

2.4.2 Groundwater Characterization

Two rounds of groundwater samples were collected from a network of seven groundwater monitoring wells installed in the Study Area, and one existing irrigation groundwater well located in OU3. Arsenic, cadmium, and lead, the constituents most frequently detected in the soil and fill material containing ash, brick, and/or glass at concentrations greater than the SCOs in the Study Area, were not detected in groundwater at concentrations greater than New York State Division of Water Technical & Operational Guidance Series standards. Groundwater monitoring to date has not identified levels of contaminants above groundwater standards.

3 ENVIRONMENTAL SETTING

OU4 comprises the southern and eastern border of the larger Study Area (Figure 1-2). The Study Area is divided into five OUs, with OU1, OU2, and OU5 all being primarily residential and OU3 being institutional (school properties). OU1, OU2, OU3, and OU5 all lie within the levy barrier constructed to reduce flood impacts from the Chemung River and Post Creek. In contrast, OU4 consists entirely of land zoned flood mitigation and public conservation land with few structures and no residences. OU4 includes an earthen flood control berm and floodplains, as shown on Figure 1-3, and serves primarily as an ecological and hydraulic buffer zone for the developed areas to the north and west. Because OU4 is a buffer with the river separated from most human activities by a levy, its vegetation, geology, soils, and hydrology are different from those of other OUs in the Study Area.

3.1 OU4 LAND USE AND LAND COVER

OU4 consists of approximately 73 acres of undeveloped land, remaining generally unchanged over time while the adjacent lands within OU1, OU2, OU3, and OU5 have been developed from farmland into a residential area and schools. Following the construction of the earthen flood control berms prior to 1901 (Rockwood 1901), only minimal activity had been conducted in OU4, primarily associated with berm construction and repair, utility construction, gravel mining, and road construction along Post Creek.

The area is primarily covered by grass and deciduous forests. U.S. Geological Survey (USGS) land cover maps classify the area land cover as pasture/hay, deciduous forest, or woody wetland (https://lta.cr.usgs.gov/GLCC). Due to the proximity of OU4 to the Chemung River and Post Creek, OU4 is designated as being within the Federal Emergency Management Agency (FEMA) 100-year and 500-year flood zones (FEMA 2002).

3.2 TOPOGRAPHY AND DRAINAGE

OU4 is generally flat, with gentle slopes to the south toward the Chemung River and east toward Post Creek. The major topographic feature in OU4 is the flood control berm that ranges from 10 to 15 ft high, and from 70 to 125 ft wide, depending on the location. The 1976 USGS 7.5-minute topographic quadrangle map for Corning, New York, indicates that the Study Area is approximately 929 ft above mean sea level (amsl). Both the Chemung River and Post Creek at base flow are generally below 910 ft in elevation with mean flows at approximately 915 ft in elevation. Within a 1 mile radius of the Study Area, ground surface elevation rises sharply toward Pine Hill to the north, and Denmark Hill to the east.

3.3 GEOLOGY AND SOILS

The Study Area is located in the Chemung River valley. Surficial geology in the Study Area includes alluvial sand and gravel adjacent to the Chemung River and Post Creek in OU4, and alluvial silt and very fine sand in the center of the Study Area (OU1, OU2, OU3, and OU5) (Miller et al. 1982). The Study Area sits in the center of a valley-fill deposit, which consists of alluvial silt, sand, and gravel, glacial-outwash, till, and lacustrine silt and clay (Miller et al. 1982) that are of post-glacial age. The river valley deposits are nearly 100 ft thick in the vicinity of the Study Area. A lens of lacustrine silts and clays has been mapped approximately 30 ft below ground surface (bgs) (Miller et al. 1982). The layer of lake silts and clay causes most of the infiltration to deeper aquifers to occur at the edges of the valley, where higher permeability exists and runoff is concentrated by the hillsides. The entire area is underlain by low permeability shale and siltstone with areas of moderate permeability due to fractures and joints (Miller et al. 1982). The geology of the region creates an alluvial aquifer in recent valley fill and deeper glacially associated silts and clays that is approximately 100 ft thick. The water levels in this alluvial aquifer are generally controlled by the elevation of the surface water bodies (Chemung River and Post Creek) as they connect hydrologically with local groundwater.

OU4 overlies predominantly sand and gravel deposits of glacio-fluvial origin, along with more recent alluvial deposits. The sand and gravel beds of OU4 have high permeabilities (Miller et al. 1982), and have a moderate infiltration potential (0.63–2.0 in. per hour) (Miller et al. 1982, Figure 2).

Soils in OU4 are mapped as fluvaquents from the earthen flood control berms to the river banks. The fluvaquents are characterized as saturated floodplain soils derived from an alluvium of highly variable texture. The berms themselves, as well as inland areas on OU1, OU2, OU3, and OU5 are mapped as Tioga silt loam. Tioga silt loam is described as deep, well-drained soils derived from loamy alluvium (USDA 2018). Given that the berms are clearly areas of historical construction, it is likely that soils on and within the berms have been modified and may vary locally.

3.4 ECOLOGICAL RESOURCES

3.4.1 Species of Interest

A review of the NYSDEC Environmental Resource Mapper shows that OU4 is in the vicinity of rare plants and rare animals (NYSDEC 2018a). The NYSDEC New York Nature Explorer identifies confirmed recent or historical distribution in the Chemung/Cohocton River watershed of the bald eagle (*Haleaeetus leucocephalus*), Henslow's sparrow (*Ammodramus henslowii*), northern harrier (*Circus cyaneus*), upland sandpiper (*Bartramia longicauda*), timber rattlesnake (*Crotalus horridus*), green floater mussel (*Lasmigona subviridis*), and brook floater mussel

(*Alasmidonta varicosa*) as state threatened species; the short-eared owl (*Asio flammeus*) as a state endangered species; and the great blue heron (*Ardea herodias)* as a state-protected bird. In addition, the NYSDEC New York Nature Explorer identifies confirmed recent or historical distribution in the Chemung/Cohocton rivers watershed of 11 state-listed endangered plant species and 6 state-listed threatened plant species (NYSDEC 2018b).

3.4.2 Wetlands

The locations of designated wetland areas in OU4 were obtained from the U.S. Fish and Wildlife Service (USFWS) National Wetlands Inventory (NWI) database (USFWS 2018). According to the NWI, areas along Post Creek are characterized as Freshwater Forested/Shrub Wetland, which includes woody vegetation over 18 feet tall and which may be temporarily flooded (from a few days to a few weeks) during the growing season. Areas of OU4 along the Chemung River are described as Freshwater Emergent Wetland habitat which may be temporarily flooded each year. This wetland type includes herbaceous hydrophytes that are present for most of the growing season and are dominated by perennial plants. The locations of the NWI-mapped wetlands are based on reconnaissance-level information, which is accurate at the nominal scale of a 1:24,000 base map, but cannot provide the accuracy of a site-specific wetland delineation. Areas within the mean high water level and wetlands are considered a potential aquatic habitat and depositional environment, and areas outside of this zone are considered a terrestrial habitat.

3.5 HYDROLOGY

3.5.1 Surface Water

OU4 and the Study Area lie within the Chemung River catchment, which is part of the larger Susquehanna River regional watershed (Figure 3-1). The Chemung River originates upstream of the City of Corning, at the confluence of the Tioga and Cohocton rivers and flows east through the Study Area. Within the Study Area, surface water flows south to southeast towards the Chemung River and Post Creek. From the confluences of Post Creek and Chemung River, flow travels southward where it merges with the Susquehanna River. The flows of Post Creek and the Chemung River have changed over time due to flood control, drainage, and infrastructure development; however, the flow path has remained generally to the south/southeast. OU4 is within FEMA-designated 100-year and 500-year flood zones (FEMA 2002).

3.5.1.1 Chemung River

The Chemung River is a tributary of the Susquehanna River, approximately 46.4 miles (74.7 km) long with a drainage area of approximately 2,006 square miles (Figure 3-1). The Chemung

River is formed near Painted Post in Steuben County, just west of the City of Corning by the confluence of the Tioga and Cohocton rivers. It flows generally east–southeast through the municipalities of Corning, Big Flats, Elmira, and Waverly. It crosses into northern Pennsylvania before joining the Susquehanna River. The Chemung River in the vicinity of the Study Area has no known use impairments and a Class C use designation (NYSDEC 2007) (Figure 3-2), which indicates the water is of high quality and is best used for fishing and is suitable for fish, shellfish, and wildlife propagation and survival, as well as for primary and secondary human contact recreation.

As part of the Susquehanna River watershed, the flow in the Chemung River is monitored at the Corning, NY USGS gaging station number 01529950, which is situated approximately 0.25 mile upstream of OU4. According to the 37-year period of monitoring, daily flows range from a minimum of 310 cubic feet per second (cfs) to 15,200 cfs, with median and mean flows of 1,100 and 2,130 cfs, respectively (USGS 2018). The mean high water level of the Chemung River and Post Creek is estimated at 915 ft amsl (Figure 3-3). This estimate is based on a mean water level of 916 ft amsl at a mean annual discharge of 2,130 cfs recorded at the gaging station, and an approximate riverbed slope of 1.2 ft/ft. The slope of the river in the vicinity of the Study Area was estimated using USGS topographic map contours in the Chemung River. This estimated mean high water level will be used as a surrogate until a field survey (see Section 5.4.3) is performed to verify the location of the mean high water level.

3.5.1.2 Post Creek

Post Creek, located along the eastern edge of OU4 is a smaller, second order stream. Post Creek extends approximately 15 miles to the northeast of the City of Corning and has a watershed area of 59 square miles. Post Creek has no known use impairments (Figure 3-2); it has a Class B use designation from the confluence with the Chemung River upstream to the top of OU4 at Pulteney Street and is Class C above Pulteney Street (NYSDEC 2007). Post Creek at OU4 is best used for primary and secondary contact recreation and fishing.

3.5.2 Groundwater

The saturated portions of the Chemung River valley deposits are recharged principally through the 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 elevation of nearby rivers (Olcot 1995). Due to the proximity of the OU4 area to the Chemung River and Post Creek and the flat topography, the groundwater table is expected to be near the ground surface in OU4 for the floodplain areas outside of the flood control berm. 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. Groundwater in the valley aquifer flows toward and discharges to nearby waterways.

3.5.3 Stormwater Outfalls

There are five stormwater outfalls within OU4, as shown on Figure 3-4. Storm drains in OU4 were constructed in 1958 and 1978, following separation of storm and sanitary sewers in the 1950s (EADS 1979). Stormwater is collected from the areas north and west of OU4 and routed to either the Chemung River or Post Creek via outlet structures through the earthen flood control berm. Three of the outfalls are located in the northeastern portion of OU4, near the OU1 boundary, and drain to a field in the direction of Post Creek (Utilities Map, City of Corning). These drain stormwater from the southern side of OU5 and the northeast area of OU5 (Utilities Map, City of Corning). Two of the outfalls are located in the southeast portion of OU4, near the Corning-Painted Post High School sports fields, and drain to the Chemung River (City of Corning 2018). These outfalls drain most of OU1, all of OU2, all of OU3, and possibly some stormwater from OU5 due to a catchment basin along E. Pulteney Street (Utilities Map, City of Corning; EADS 1979), as well as areas to the west of the Study Area. Note that one outfall observed in the southeast portion of OU4 is not shown on the City of Corning Utilities Map; the current status of the outfall will be verified during the proposed activities.

3.6 REGIONAL CONDITIONS

Within the Study Area, OU4 is the most subject to regional influence due to episodic flooding events that bring soil and sediment from upriver, as well as its location at the downstream end of several stormwater drainage areas, including Post Creek. Soil and sediment that may erode from OU4 will mix with sediment from upstream and downstream of the Study Area as they are transported downriver. Any impacts to sediment or surface water adjacent to or downstream of OU4 could originate from a number of potential sources in the region, and an understanding of regional conditions provides a context for understanding observed conditions in OU4.[1]

Recent studies of regional water quality and biology consistently found a healthy and unimpaired ecosystem in the Chemung River in the vicinity of OU4 (Figure 3-2) (NYSDEC 2004, 2007). The NYSDEC water quality assessment reported no known use impairments for the Chemung River based on benthic macroinvertebrate sampling conducted periodically along different reaches of the Chemung River from 1973 to 2002. Benthic macroinvertebrate samples were collected along 5-meter increments within riffles using a traveling kick method, where an aquatic net was positioned downstream of the sampler, the stream bottom was disturbed by foot, and dislodged organisms were carried and captured in the net. Species richness (number of species or taxa found in a sample), EPT value[2] (number of mayflies, stoneflies, and caddisflies in a sample), Hilsenhoff Biotic Index (average tolerance value for all organisms in a sample),

[1] For example, if contaminants are observed in the sediment downstream of OU4 that are not observed in OU4, the conceptual site model would conclude that they did not originate in OU4.
[2] EPT index is the relative abundance of Ephemeroptera, Plecoptera, Trichoptera orders.

and percent model affinity (measure of similarity to a model non-impacted community) were used to determine the levels of water quality impacts in rivers and streams (NYSDEC 2004).

Macroinvertebrate diversity was also used in impact source determination to identify the types of impacts that exert deleterious effects on a waterbody. Water quality issues identified downstream of the Study Area were related to pathogens and pesticides in the most downstream segment of the Chemung River (Waverly to Wellsburg). Upstream of the Chemung River, in the Tioga and Cohocton watersheds, impairment in some stream segments has been identified for silt and sediment (due to bank erosion), dissolved oxygen, and nutrients.

The Susquehanna River Basin Commission performed a biological community survey with five sites located along the Chemung River downstream of OU4 in June/July 2012 (Campbell 2013). Results from biological sampling found the site located immediately downstream of the confluence with Post Creek "…had a nonimpaired biological community, excellent habitat, and higher water quality" (Campbell 2013) (see Figure 3-2). Results from a second location farther downstream found "…slightly impaired biological community, excellent habitat, and middle water quality from elevated total sodium and the highest concentration of total bromide observed within the study" (Campbell 2013). The remaining three sites along the Chemung River were located farther downstream from OU4.

Biological monitoring was conducted along reaches of Post Creek in the vicinity of OU4 in 1998 and 2002 (Figure 3-2) (NYSDEC 2004, 2007). Results of stream biomonitoring along Post Creek, both upstream and within the OU4 boundary (NYSDEC 2004), are as follows:

> Non-impacted water quality was assessed for the site in Corning, based on macroinvertebrate sampling in 1998 and 2002. A diverse and well-balanced fauna was found, including clean-water mayflies, stoneflies, riffle beetles, and hellgrammites.

Biological community survey sampling at two sites on Post Creek in 2012 found a slightly impaired biological community at both the upstream location (due to poor riparian conditions) and the downstream location (Figure 3-2), but the downstream location "…had excellent habitat and water quality" (Campbell 2013).

4 CONCEPTUAL SITE MODEL

A conceptual site model (CSM) was developed for OU4 based on site-specific, historical, and regional data and information. The CSM is a tool that provides a context for understanding historical, present, and potential future site conditions, including potential conditions that may or may not result in exposure, and a structure for developing an appropriate data collection program for site characterization. The CSM also provides a framework to interpret and holistically consider all of the data available. The CSM will be refined, as appropriate, as new data are collected.

The individual elements of the CSM have been developed based on available data, including visits, aerial photos, historical soil borings, data from other OUs in the Study Area, regional ecological and sediment data, and application of basic physical and chemical fate and transport processes. These elements are integrated in the CSM to develop a cohesive understanding of OU4.

4.1 EVIDENCE OF FILL

The available data and information, including historical aerial photos and borrow pit and coring log observations, do not suggest obvious areas of fill within OU4, except in areas near the boundaries of OU2 and OU3.[3] While these data cannot provide a complete historical characterization of potential fill locations, they provide a basis for identification of data gaps and focus for data collection activities. If any fill is found in OU4 downgradient of the flood control berm, the potential sources of fill are likely:

- Intentional placement—For example construction spoils from berm construction or Post Creek relocation activities. Given that OU4 is within the floodplain, fill placement for flood control (specifically to elevate house lots above the floodplain) that took place in the upland OUs would not have been performed in OU4.
- Berm damage—Fill material may have been transported out of the upland OUs and deposited in OU4 during the 1972 Tropical Storm Agnes, when the berm was damaged.
- Stormwater—Fill material may have been historically transported out of the upland OUs or from outside of the Study Area with stormwater and deposited at the stormwater outfalls.

USACE produced an "as-built" report as part of the construction of the flood control berm (USACE 1941). As discussed above (see Section 2.2.2), there was fill observed in 2 of the 17 cores within OU4 (FP-18 and FP-20), and one location (FP-20) where the fill was identified

[3] Including areas depicted by the photographs provided by NYSDEC on May 18, 2020, which indicate the presence of ash, brick, and glass at the east end of Corning Boulevard.

specifically as "cinders, glass, etc." This core was located where the berm was built, and fill material in this area would have been excavated during the construction of the berm foundation. The report also identified one small area within OU4 as "Fill (Spoil Material)" along the Post Creek side of the berm. Based on the data presented in this report, there were limited, if any, areas of fill placement prior to 1941.

Historical aerial photos were reviewed with a specific focus on evidence of construction or disturbance activities that may have resulted in placement of fill (see Section 2.2.1). The series of photos shows that the floodplain has been largely stable over time, evidenced by the mature forested area along Post Creek and the unchanged location of the upper portion of the dirt road into OU4 in the southwest corner. Temporal changes observed in the sequence of photos include repositioning of the lower portion of the dirt road (particularly following Tropical Storm Agnes) and relocation of Post Creek in 1950s and early 1980s. The aerial photos show that OU4 has been stable for decades and do not provide strong indication of areas of fill within OU4.

4.2 FATE AND TRANSPORT PATHWAYS

The pathways for potential transport include stormwater from upland OUs and outside of the Study Area and sediment transport and deposition during high flow and flood events. To date, no pathways for fill material to enter OU4 downgradient of the flood control berm from upland areas have been established.[4]

The City of Corning stormwater system includes five stormwater outfalls within OU4 (Figure 3-4). The storm drain in the northern part of OU4 at the end of E. Pulteney Street drains a very small area and appears to drain into the outfall at the end of Argonne Street. The outfalls drain upland areas including the Study Area and adjacent neighborhoods to the north and the west of the Study Area boundaries (Figure 3-4). Given that the ground surface within the Study Area is predominantly paved or vegetated, and that the stormwater drainage area extends beyond the Study Area boundary, current stormwater quality is anticipated to reflect urban background conditions.

OU4, which is within the 100-year floodplain of the Chemung River and Post Creek, is subject to periodic flooding. Floodwaters have the potential to erode and deposit sediment in OU4. Examination of historical aerial photos indicates that the floodplain is stable overall (see Section 2.2.1). The periodic inundation would have the potential effect of reworking surficial sediment and depositing sediment originating from upstream to OU4. It is anticipated that soil and sediment subject to periodic inundation would reflect regional sediment quality, and the

[4] This Work Plan identifies potential pathways; the results of the characterization will serve to confirm or deny these pathways.

more frequently an area is subject to inundation, the stronger the regional background conditions are expected to influence sediment quality.

4.3 POTENTIAL EXPOSURE PATHWAYS

Potential exposure pathways include the receptors that could be exposed to fill material in OU4 and the pathways through which the exposure could occur. Overall, thick vegetation with grasses exists over much of the area along Chemung River and with mature forest along Post Creek, which serves to limit, and likely eliminate, potential exposure to underlying soil and any fill that may be present in the surface or subsurface soil.

4.3.1 Human Health Exposure Scenarios

Likely human use of OU4 is limited to occasional visitation, and includes maintenance (workers who occasionally mow the grass) and recreational activities (e.g., dog walking). OU4 is entirely within the 100-year floodplain, which precludes residential use.

Potential exposure pathways are direct contact, incidental ingestion, and/or inhalation of soil and direct contact and incidental ingestion of sediment. Based on the extent of the vegetation, the soil pathways are largely incomplete; that is, if there are areas of fill material in the soil in OU4 downgradient of the flood control berm, there is no exposure occurring (Figure 2-6). Exposure to sediment could occur in limited areas along the Chemung River and Post Creek where banks are periodically inundated with floodwaters, preventing establishment of stable vegetation (Figure 2-7); however, water access is limited in portions of OU4 due to steep banks and dense vegetation. As discussed above, these riverbanks are largely influenced by regional fate and transport processes. Potential human health exposure scenarios will be defined following characterization and will be subject to review and approval by NYSDEC and the New York State Department of Health (NYSDOH).

4.3.2 Ecological Exposure Scenarios

The primary focus of ecological exposure is on upland receptors that may be present in OU4, including plants, soil invertebrates, deer, groundhogs, raccoons, field mice, songbirds, and raptors. Sediment exposures could include benthic invertebrates, fish, and other wildlife that may inhabit these waterbodies. Potential ecological exposure scenarios will be defined following characterization and will be subject to review and approval by NYSDEC and NYSDOH.

4.4 REGIONAL CONTEXT

An important aspect in interpretation of observed conditions is understanding regional conditions and local impacts. As described in Section 3.6, OU4 is subject to regional influence due to episodic flooding events that bring soil and sediment from upriver, as well as its location at the downstream end of several stormwater drainage areas, including Post Creek. It is critical to distinguish this regional influence when characterizing OU4, simply because conditions within OU4 may originate elsewhere.

4.5 SUMMARY

The OU4 CSM is summarized below, and important features that support the CSM are presented in Figure 4-1.

- The potential exposure pathways in OU4 differ from those in other OUs in the Study Area; most importantly there is no residential use in OU4.
- Potential fate and transport into and out of OU4 is part of the regional system, and soil and sediment quality in OU4 must be evaluated within a regional context.
- There is little to no evidence of fill in OU4, evidenced in the historical data collected to support berm construction and historical aerial photos.
- Locations where ash, brick, and glass have been observed are on the upland side of the flood control berm.
- There are limited means for fill to be present within OU4, given the historical and current use and lack of significant construction activities.
- The ecosystem is healthy, indicated by a mature forest, dense grasses, the presence of healthy ecological indicators, and healthy regional conditions.
- Regional water quality is deemed generally good, with some silt/sediment, nutrients, and pesticides impairments that are unrelated to the Study Area.
- Regional influences could have an impact on water and sediment quality in the Chemung River and Post Creek.

5 CHARACTERIZATION ACTIVITIES

Characterization in OU4 will be performed in a phased approach (Section 5.1), and includes surveying activities and sampling activities. Surveying activities (Section 5.4) will be completed prior to initiation of the sampling activities (Section 5.5).

5.1 PHASED FIELD INVESTIGATION

The OU4 characterization will be performed in a phased manner and will include multiple field mobilizations, as necessary, to achieve the objectives of the characterization. Phase 1 investigation activities include soil sampling at locations where historical disturbance has been identified in the historical document and aerial photo review, where transport into the OU may have occurred, to confirm historical sampling results (i.e., results presented in USACE [1941]), and to provide spatial coverage. The approximate sampling locations are depicted on Figure 5-1, and a summary of location selection rationale is presented in Table 5-1; however, the final number and locations will be determined prior to sampling based on conditions encountered in the field, in accordance with the NYSDEC Screening and Assessment of Contaminated Sediment guidance document (2014), and as agreed upon by NYSDEC.

Phase 2 investigation activities include sediment and surface water sampling at locations within OU4 to be finalized based on Phase 1 results and the NYSDEC-approved mean high water level (see Section 5.4.3), as approved by NYSDEC. Following completion of Phase 1 investigation activities, the validated data will be reviewed to refine the CSM and to inform the design of the Phase 2 activities. Conceptual sediment/surface water sampling locations are presented in Figure 5-1.

Surface water samples will also be collected at the locations of the five storm drains, collocated with the soil samples at these locations (Figure 5-1). These samples will be collected opportunistically, when flow through the storm drains occurs, and could be collected in either Phase 1 or Phase 2.

5.2 LIMITS OF INVESTIGATION

The OU4 characterization sampling includes collection of surficial and subsurface soil borings, and sediment and surface water samples within OU4. Soil sample locations in Phase 1 are generally located above mean high water level (see Section 3.5.1), where regional influences are less likely to confound the findings. Locations were selected to characterize stormwater outfalls. Note that these locations were selected to characterize any potential impact from upland stormwater runoff to OU4, and will be evaluated as appropriate, following completion of the biological survey delineating the mean high water level, consistent with other samples

collected during the field investigation. Use of the mean high water demarcation provides a boundary between terrestrial (soil) and aquatic (sediment) habitat. Phase 2 will include sample locations below mean high water level, within the boundaries of OU4.

The Remedial Order excludes the berm and a buffer area around the flood control berm. Accordingly, no investigation or remediation work will be performed within the footprint of the constructed earthen flood control berms including a buffer area from the toe of the earthen flood control berm, referred to as the berm exclusion area (Figure 5-1). The width of the berm exclusion area will be determined based on the estimated extent of the berm, using a 2.5 ft horizontal to 1 ft vertical slope from the toe of the slope.

5.3 WRITTEN ACCESS CONSENT

Property within OU4 is not owned by, or under the control of, Corning or NYSDEC. The work will be performed under an access agreement between Corning and the property owner.

5.4 SURVEY ACTIVITIES

Prior to sample collection, surveys will be performed to confirm and mark the boundaries of OU4, the wetlands within OU4, and the mean high water level along the Chemung River and Post Creek.

5.4.1 OU4 Boundary Surveying

The OU4 boundaries and the peak and toe of the slope of the flood control berm[5] will be surveyed prior to the initiation of work by a professional land surveyor licensed in the State of New York and submitted to NYSDEC for review prior to finalization of the sample locations.

5.4.2 Wetland Delineation

A wetland delineation will be performed by a qualified Environmental Scientist to establish the boundaries between terrestrial and aquatic habitats within OU4 under current conditions. The delineation will be performed in accordance with the requirements specified in the New York State Freshwater Wetlands Delineation Manual (NYSDEC 1995). The boundary of the delineated wetlands will be recorded using a handheld global positioning system (GPS) unit with submeter accuracy.

[5] The berm exclusion area will be established based on the survey data.

5.4.3 Biological Survey

A biological survey will be performed to confirm or refine the mean high water level. The survey includes identification of plant species that live in aquatic habitat to identify the boundary between sediment and soil.[6] Appendix F presents the approach to the Biological Survey.

5.5 SAMPLING ACTIVITIES

The subsections below include descriptions of the approaches and field investigation methodologies to be used for the characterization of OU4. The methodologies may be adjusted in the field based on a variety of factors, including field conditions, selected contractor equipment availability, and other necessary adjustments. NYSDEC will be notified of any proposed substantial changes or deviations from the approved OU4 Characterization 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 for their verbal concurrence, confirmed by subsequent email and/or documented in the field notes, and noted in the investigation summary report. Final locations will be established based on utility clearance and accessibility. Sample collection procedures are provided in the SOPs in Appendix D.

5.5.1 Surface and Shallow Soil Sampling

5.5.1.1 Sampling Locations

Nine surface and shallow soil samples will be collected at the approximate locations shown on Figure 5-1. The rationale for each sample location is shown on Table 5-1. All sampling locations will be recorded using a handheld GPS unit with submeter accuracy.

5.5.1.2 Sampling Methods

Surface soil samples will be collected for analysis from 0 to 6 in. bgs, excluding the vegetative cover or sod layer. Shallow soil samples will be collected from 6 to 12 in. bgs and 12 to 24 in. bgs, excluding the vegetative cover or sod layer. Prior to sample collection, visible vegetative matter (i.e., sod layer) will be removed. Surface soil and shallow soil samples will be collected using a small direct-push drill rig or a handheld steel soil auger, following the procedures described in SOP SL-07. Soil sampling methodologies will be employed that ensure that the soil structure will be retained to a 1-in. interval to identify ash, brick, and/or glass, if present. The

[6] As stated in New York State Regulation 6 CRR-NY 608.1(r), vegetative characteristics are one line of evidence, along with hydrologic data and physical characteristics, to establish the mean high water level.

soil will be described, noting the color, moisture content, texturing, layering, evidence of disturbance (foreign debris), and the distribution/abundance of roots and screened with a photoionization detector (PID). If present, layers of fill material containing ash, brick, and/or glass will be noted in the field logs. Surface and shallow soil samples will be homogenized (for analyses other than volatile compounds) and placed directly into appropriate laboratory-prepared sample containers.

Surface and shallow soil sample will be analyzed for target analyte list (TAL) metals plus mercury, and target compound list SVOCs. A subset of the surface and shallow soil samples (20 percent) will be analyzed for per- and polyfluoroalkyl substances (PFAS), and 1,4-dioxane. The analytical methods and protocols to be used during this project as well as the expanded list of constituents for analysis are provided in the QAPP (Appendix C), Tables C2-1 and C2-3, respectively.

Surface soil and shallow soil sample locations will be recorded using a handheld GPS unit with submeter accuracy. All nondedicated sampling equipment will be decontaminated by washing with phosphate-free detergent and rinsing with distilled water prior to and between sampling locations, as described in SOP SL-01.[7] Alternatively, dedicated, disposable sampling equipment (e.g., scoops, plastic blending trays) may be used. Decontamination fluid handling and disposal is described in Section 5.7.

Soil samples and appropriate quality control samples (e.g., duplicate samples) will be placed in appropriate laboratory-prepared containers in iced coolers and shipped with completed chain-of-custody documentation to TestAmerica Laboratories, Inc. in Buffalo, New York (TestAmerica) for analysis, following the protocols in SOP SL-02, SOP AP-01, and SOP AP-03.

5.5.2 Soil Boring Sampling

5.5.2.1 Sampling Locations

Twenty-one soil borings will be advanced at the approximate locations identified on Figure 5-1. The rationale for each sample location is shown on Table 5-1. All sampling locations will be recorded using a handheld GPS unit with submeter accuracy.

5.5.2.2 Sampling Methods

Soil borings will be advanced to a depth up to 8 ft bgs, or until refusal. In soil borings where no layer of fill material containing ash, brick, and/or glass is encountered, up to three samples will be collected per boring: 0 to 6 in. bgs (excluding the ground cover or sod layer), and two

[7] Solvents will not be used unless materials are encountered in the field that cannot be cleaned with detergent and water.

shallow soil samples from 6 to 12 in. bgs and 12 to 24 in. bgs, if present above refusal. In a subset of the locations (20 percent), an additional sample will be collected from the native material or the bottom of the boring.[8] Samples will be analyzed for TAL metals plus mercury and SVOCs. A subset of the soil boring samples (20 percent) will be analyzed for PFAS and 1,4-dioxane.

If a layer of fill material containing ash, brick, and/or glass is encountered in a floodplain soil boring, up to five soil samples will be collected per boring: 0 to 6 in. bgs (excluding the ground cover or sod layer), and two shallow soil samples from 6 to 12 in. bgs and 12 to 24 in. bgs, or to the depth of the layer of fill material containing ash, brick, and/or glass; one from the layer of fill material containing ash, brick, and/or glass; and one from the native material beneath the layer of fill material containing ash, brick and/or glass, if present.

Soil boring samples will be analyzed for TAL metals plus mercury and SVOCs. A subset of the soil boring samples (20 percent) will be analyzed for PFAS and 1,4-dioxane. The analytical methods and protocols to be used during this project as well as the expanded list of constituents for analysis are provided in the QAPP (Appendix C), Tables C2-1 and C2-3, respectively.

Generally, either direct-push (Geoprobe® or equivalent) or hollow-stem auger drilling technologies will be used to advance soil borings to characterize the subsurface soils, using the procedures described in SOP SL-07. Where possible, direct-push drilling technology will be utilized to minimize the quantity of IDW generated during field activities. A hollow-stem auger drill rig will be used to install borings in locations where direct-push cannot penetrate to the desired depth (up to 8 ft bgs). All drilling locations will be cleared of buried utilities prior to drilling using various geophysical methods and the New York State One-Call system. Soil boring locations will be recorded using a handheld unit interfaced with a GPS unit with submeter accuracy.

At each boring location, soil sampling will be conducted on a continuous basis (if possible) from the ground surface to the maximum sampling depth or refusal, using a 2-in., 4-ft-long macrocore sampler. Retrieved soil samples will be examined in the field for physical description by a qualified geologist and screened with a PID. Layers of fill material containing ash, brick, and/or glass will be identified in the field by the geologist. Field observations and descriptions of soil cores will be documented as described in Section 6.3. A layer of fill material is defined as a non-native material containing ash, brick, and/or glass with a thickness of greater than 1 in. All direct-push rods and associated drilling equipment will be cleaned between boring locations using the procedures described in SOP SL-01 (Appendix D).

Where hollow-stem auger drilling technology is used, hollow-stem augers will be extended from ground surface to the desired depth. Samples will be continuously collected with a 2-ft-

[8] Sampling of native material will be prioritized in borings that contain ash, brick, and/or glass above the native material.

long split-spoon sampler during drilling for physical description in the field by a qualified geologist and screened with a PID. Layers of fill material containing ash, brick, and/or glass will be identified by the geologist. All hollow-stem augers and associated drilling equipment will be cleaned between boring locations using the procedures described in SOP SL-01 (Appendix D).

Samples will be visually examined and a description prepared by a qualified geologist in accordance with the procedure described in SOP SL-04 (Appendix D) and logged in accordance with the procedures described in SOP SL-04. The description will generally be prepared using the Unified Soil Classification System (USCS) (ASTM D2487; ASTM 2017a), and will include color, moisture content, texture, layering, etc. Any non-native material present in the sample will be noted and described (type, color, texture, moisture content, etc.) and any layer of fill material containing greater than 1 in. of ash, brick, and/or glass will be noted in the field logs. Field observations and descriptions of the collected samples will be recorded in the field logbook or soil boring log form. Photographs of the soil cores will be taken.

All nondedicated sampling equipment will be decontaminated prior to and between sampling locations. Decontamination procedures are described in Appendix D (SOPs), and decontamination fluid disposal is described in Section 5.7. Alternatively, dedicated, disposable sampling equipment (e.g., scoops, plastic blending trays) may be used.

Soil samples and appropriate quality control samples (e.g., duplicate samples) will be collected from the sampling cores, homogenized in the field, placed in appropriate sample containers in iced coolers, and shipped with completed chain-of-custody documentation to TestAmerica for analysis.

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., topsoil, sod, and grass seed). IDW from this investigation will be contained in sealed containers (e.g., drums or other appropriate containers) and staged in a secondary containment area at the designated location, as described in Section 5.7.

5.5.3 Sediment Sampling

5.5.3.1 Sampling Locations

Sediment samples will be collected in the Chemung River and Post Creek within OU4. Conceptual sample locations are shown in Figure 5-1. Sampling locations will be adjusted based on the results of the Phase 1 soil sampling and the refined CSM, as approved by NYSDEC. Sampling locations will be recorded using a handheld GPS unit with submeter accuracy.

5.5.3.2 Sediment Core Collection

Sediment samples will be collected using either handheld or boat-based methods, depending on access and water depth. Multiple samples may be collected at each location, as necessary to achieve the required sample volumes.

Shallow water sediment cores will be collected using a handheld sediment coring device consisting of a core tube with an attached slide-hammer (or similar device), in accordance with SOP SD-14 (or equivalent equipment). The core tube with slide-hammer is advanced by repeatedly hitting the top of the core with the weighted hammer until the desired sampling depth has been achieved. A Lexan™ or polyethylene core tube will be used as the core tube. Handheld sediment cores will be advanced to a maximum depth of 2 ft. Sediment cores in deeper water will be collected using a vessel-deployed vibracore system (or equivalent technology). Vessel-deployed sediment cores will be advanced to a maximum depth of 2 ft. Vibracorers generally consist of a metal corer barrel (usually a 4-in.-outside-diameter, aluminum core barrel) with a location-dedicated polycarbonate or Lexan™-lined core tube, and a vibrator mechanism attached to the top of the barrel. The vibration is created either by an electric motor, a hydraulic system, or a pneumatic piston attached to the top of the barrel. A continuous sediment sample is retained within the tubing with the aid of a core cutter/core catcher attached to the bottom of each tube. Vibracore collection methods will follow standard protocols and guidelines as described in the SOP SD-08 (Appendix D).

Sediment core samples will be collected from 0–6, 6–12, and 12–24 in. intervals. Refusal may be encountered in less than 2 ft; however, the sample will be considered acceptable if sediment recovery is greater than 75 percent. The coordinates of sample locations will be recorded using a handheld digital global positioning system (DGPS) with submeter accuracy.

Before sampling begins, core tubes will be decontaminated following procedures outlined in SOP SD-01.[9] During storage and transport, empty decontaminated core tubes will be capped at both ends to prevent possible contamination. Once the sediment core is collected and returned to the sample processing area, the overlying water will be siphoned from the top of the core. Both ends of the core will be securely capped; labeled with the station identifier, core section interval, and sediment orientation; and secured in an upright position. Field quality control samples will be collected in accordance with SOP SD-02.

Sediment cores will be continuously collected and logged using ASTM D2488 guidelines (ASTM 2017b) and following SOP SD-08 (Appendix D). Core lithology, sample IDs, and sample depth intervals will be recorded on the core log. The core log form will be completed in accordance with SOP AP-02 and will include the following information:

[9] Solvents will not be used unless materials are encountered in the field that cannot be cleaned with detergent and water.

- Core penetration depth and recovery
- Physical sediment description (i.e., sediment classification, density/consistency, color)
- Odor (e.g., hydrogen sulfide, petroleum)
- Visual stratification and lenses
- Vegetation
- Presence of debris (natural or anthropogenic objects)
- Presence of oily sheen or obvious contamination
- Evidence of biological activity (e.g., detritus, shells, tubes, bioturbation, live or dead organisms)
- Other distinguishing characteristics or features.

Upon completion of the core description log, the cores will be processed following the procedures described in the following section.

5.5.3.3 Sediment Sample Processing

Samples will be homogenized by depth intervals (i.e., 0–15, 15–30, and 30–60 cm [0–6, 6–12, and 12–24 in]). Samples for analysis of sulfides will be sampled directly from each depth interval prior to sample homogenization. Sediments from each depth interval will be placed in a decontaminated stainless-steel bowl and thoroughly mixed to a uniform color and texture. The sediment will be stirred periodically while individual aliquots for analyses are taken to ensure that the mixture remains homogenous. All excess sediment will be stored and managed in accordance with procedures outlined in Section 5.7 and SOP AP-05. All decontamination fluids will be managed in accordance with procedures outlined in SOP AP-05 (Appendix D).

The analyte list for the sediment samples, including ancillary parameters that are associated with potential bioavailability, is:

- TAL metals
- Mercury
- SVOCs
- Total organic carbon (TOC)
- Grain size
- Sulfides
- Nitrates
- Carbonates

- Ancillary parameters to consider for understanding of bioavailability (pH, redox [field and lab measured], and cation exchange capacity)
- A subset of the sediment samples (20 percent) will be analyzed for 1,4-dioxane, and PFAS.

The analytical methods and protocols to be used during this project as well as the expanded list of constituents for analysis are provided in the QAPP (Appendix C), Tables C2-1 and C2-3, respectively.

Each sample collected in the field will be labeled in accordance with the Standard Operating Procedure for Sampling Labeling (SOP AP-04, Appendix D). Samples will be managed according to the protocols described in SOP AP-01, Standard Operating Procedure for Sample Packaging and Shipping (Appendix D), and SOP AP-03, Standard Operating Procedure for Sample Custody (Appendix D), and the QAPP (Appendix C). Field activities will be documented in accordance with SOP AP-02 (Appendix D).

5.5.4 Surface Water Sampling

Surface water samples will be collected at the five stormwater outfalls and at locations collocated with the sediment samples, as described below.

5.5.4.1 Sampling Locations

Surface water samples will be collected from the five stormwater outfalls, collocated with the soil sampling locations (Figure 5-1). Sampling locations will be recorded using a handheld GPS unit with submeter accuracy.

Surface water samples will also be collected in the Chemung River and Post Creek within OU4, collocated with the sediment samples.[10] Conceptual sample locations are shown in Figure 5-1. Sampling locations will be adjusted based on the results of the Phase 1 soil sampling and the refined CSM, as approved by NYSDEC. Sampling locations will be recorded using a handheld GPS unit with submeter accuracy.

5.5.4.2 Sampling Methods

Before sampling, equipment will be decontaminated following the protocols in SOP SW-01.[11] Surface stormwater sample collection will involve obtaining representative samples of the outfall water using a clean sample-collection bottle and submersing the sample bottle in water,

[10] Surface water samples will be collected at sediment sampling locations where water depth of 6 in. or greater is present.
[11] Solvents will not be used unless materials are encountered in the field that cannot be cleaned with detergent and water.

mouth pointed upstream and below the water surface using a sampling pole (e.g., swing sampler) in accordance with SOP SW-20. Samples for dissolved constituent analysis will involve collecting the sample in a new, laboratory-supplied unpreserved sample bottle, and then transferring the water to a hand pump filtration apparatus for field filtration. Filtered samples will be obtained using a disposable 0.45-µm cartridge filter. After filtration, the water will be transferred to the appropriate sample bottles.

Chemung River and Post Creek surface water samples will be collected as grab (not composite) samples and generally following sampling techniques described in SOP SW-04. Samples will be collected from downstream to upstream. Filtered and non-filtered samples will be collected using a portable peristaltic pump. Tubing leading to pump inlet will be placed in the flowing water portion of the stream and elevated as needed to avoid potential uptake of stream bed sediments. Sample containers specific to each analysis will be held near the pump outlet and then filled. For filtered samples, a 0.45-µm disposable filter will be placed in-line at the tube outlet to filter samples immediately before the water is discharged into the sample container. The coordinates of sample locations will be recorded using a handheld DGPS with sub-meter accuracy following procedures outlined in SOP AP-06 (Appendix B).

In situ water quality parameters (pH, temperature, and oxidation-reduction potential [ORP]) will be collected for all surface water sample locations. Surface water samples will be analyzed for TAL metals (total and dissolved), mercury, total suspended solids, total dissolved solids, TOC, dissolved organic carbon, and SVOCs. A subset of the surface water samples (20 percent) will be analyzed for PFAS, and 1,4-dioxane. The analytical methods and protocols to be used during this project as well as the expanded list of constituents for analysis are provided in the QAPP (Appendix C), Tables C2-1 and C2-3, respectively.

Each sample collected in the field will be labeled in accordance with the Standard Operating Procedure for Sampling Labeling (SOP AP-04, Appendix D). Samples will be managed according to the protocols described in SOP AP-01, Standard Operating Procedure for Sample Packaging and Shipping (Appendix D), and SOP AP-03, Standard Operating Procedure for Sample Custody (Appendix D), and the QAPP (Appendix C). Field activities will be documented in accordance with SOP AP-02 (Appendix D).

5.6 QUALITY ASSURANCE/QUALITY CONTROL

To ensure quality throughout the project, trained and experienced personnel will be assigned appropriately, and SOPs and approved analytical methods will be employed for sample collection, preservation, analysis, and documentation. In addition to the laboratory quality assurance and quality control (QA/QC) samples analyzed in accordance with the laboratory QA/QC Plan, several field quality control samples will be collected and submitted for analysis

throughout the course of the field investigation, to assess the quality of data obtained from the field sampling program. The quality control samples include:

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

The number of QA/QC samples anticipated is tabulated in QAPP Table C2-2. Further descriptions of the QA/QC samples and analytical procedures are provided in the QAPP (Appendix C).

Laboratory data deliverable packages will meet the requirements of NYSDEC Analytical Services Protocol Category B (see DER-10 Appendix 2B, Section 1.0b). Validation of laboratory data deliverable packages will be performed as described in Section 6.3.4.

5.7 WASTE HANDLING

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

6 PROJECT MANAGEMENT

The following sections describe the schedule for implementing the work plan and the management of data and information generated for characterizing OU4.

6.1 ROLES AND RESPONSIBILITIES

The OU4 characterization activities will be performed by a qualified contractor(s) on behalf of Corning. It is anticipated that this work, consistent with the activities performed to date in the Study Area, will be performed under the oversight of a NYSDEC representative. Contact information for communications between Corning and agency representatives is provided in the Remedial Order. Contact information for Integral has been provided to NYSDEC.

6.2 SCHEDULE

The activities described in this OU4 Work Plan are expected to be performed following NYSDEC approval of this work plan and upon receipt of consent to access from the property owner. Sampling activities are anticipated to occur between June and September during a period of low seasonal flow conditions in the Chemung River and Post Creek. The anticipated project schedule is provided as Figure 6-1. The schedule will be updated as needed and submitted to NYSDEC and NYSDOH.

6.3 DOCUMENTATION

Essential project information related to field sampling and data analysis will be documented in logs and reports, which will be retained by Corning and/or their contractor(s). Corning will submit an OU4 characterization report to NYSDEC following completion of the OU4 investigation.

6.3.1 Field Logs

Project information pertinent to field activities, including sampling, will be recorded in bound field logbooks with consecutively numbered pages and/or field data forms specific to a given activity. Information recorded in the field logbook will contain a variety of information such as:

- Date and time of logbook entry
- Names of all field personnel
- Weather conditions

- Field observations/measurements
- Ambient air monitoring data
- Summary of daily activities and significant events
- Description of samples and sampling locations
- Date and time of sample collection
- Collector's sample identification number(s) and/or name
- Name and affiliation of visiting personnel
- Decontamination activities
- Description of any problems or issues encountered and resolution
- Description of any deviations from this work plan.

Entries will be made in ink with no erasures. If an incorrect entry is made, the information will be marked with a single strike line, initialed, and dated. At regular intervals (e.g., daily or weekly), field staff will create scanned or photographed electronic copies of the field logbook pages and field data forms. Recording requirements outlined in the COVID-19 Addendum to the HASP will be followed, consistent with New York State and U.S. Centers for Disease Control and Prevention protocols.

6.3.2 Photograph Log

A project photograph log will be prepared and maintained by the contractor(s) throughout the characterization activities to provide photograph documentation of field activities. In particular, photographs of each sample location and of the soil boring cores will be collected, logged, and retained.

6.3.3 Field Reports

Field contractors will prepare brief daily work activity reports summarizing the work performed each day. At the completion of the project, all documents will be provided to Corning. During the execution of the work described in this OU4 Work Plan, field contractors will periodically provide NYSDEC and NYSDOH representatives with verbal updates of the field activities, as well as electronic copies of work activity reports with supporting photographs. All ambient air monitoring data recorded in the field logbook or designated field sheets will be communicated by the field contractor to NYSDEC and NYSDOH on a scheduled basis (i.e., daily for levels which require actions, weekly for routine monitoring data).

6.3.4 Data Management

Field measurements and laboratory analytical data will be managed by contractors in an electronic database and will be uploaded in an electronic data deliverable (EDD) format.

Laboratory data deliverable packages will be reviewed for completeness, adherence to holding times, consistency with chains-of-custody, and consistency with planned analytical methods. A qualified contractor will perform data validation and prepare DUSRs in accordance with procedures described in the QAPP (provided in Appendix C).

6.3.5 Reporting

Upon receipt of validated data, Corning will supply such validated data to NYSDEC in DUSRs. The DUSRs will include tables summarizing sample analytical results as well as tables summarizing QA/QC sample results. NYSDEC will review the data and Corning's transmittal letters before they are provided to individual property owners.

Following the completion of characterization activities, and upon NYSDEC acceptance of the DUSRs, an OU4 characterization report documenting the investigation and findings will be prepared and submitted to NYSDEC. This report will include a summary of all activities, including a description of any deviations from the Work Plan, as well as the submission of analytical results including the results of the QA/QC samples.

The OU4 characterization report will contain the following information:

- Summary tables of field and laboratory analytical data
- Maps and/or aerial photographs showing soil sampling locations
- Photo logs
- Soil boring logs
- Comparison to appropriate soil cleanup objectives
- Discussion of sampling results and significant findings.

6.4 HEALTH AND SAFETY PLAN

The health and safety of field personnel, clients, visitors, and the community are of utmost importance. It is anticipated that Level D personal protection (i.e., coverall or work clothes, work boots, safety glasses, and hard hat) will be utilized by workers during characterization activities. All field activities will be conducted in accordance with the HASP and CAMP provided in Appendix A and Appendix B, respectively. An addendum to the HASP provides COVID-19 protocols.

6.5 STUDY AREA CONTROLS

A temporary field office and equipment storage area are currently established in a NYSDEC-approved staging area near the Study Area. This temporary field office area is surrounded by temporary fencing for security and the access gate is closed and locked when not in use. The temporary field office consists of an office trailer for document and sample preparation, and staging area for field equipment. Electricity is supplied to the mobile office via a power drop.

6.6 COMMUNITY RELATIONS

A supplemental Citizen Participation Plan for the Study Area has been prepared in accordance with DER-10 requirements and submitted to NYSDEC (Corning 2014). The Citizen Participation Plan describes the community relations components to be followed during the implementation of this OU4 Work Plan and other Study Area related activities.

7 REFERENCES

ASTM. 2017a. ASTM D2487, Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System). ASTM International, West Conshohocken, PA.

ASTM. 2017b. ASTM D2488, Practice for Description and Identification of Soils (Visual-Manual Procedures). ASTM International, West Conshohocken, PA.

Campbell, E. 2013. Chemung River Subbasin - Year-1 Survey. Publication 287. Susquehanna River Basin Commission. September.

City of Corning. 1936. Record of Regular Monthly Meeting of Honorable Mayor and Members of the Common Council of the City of Corning, New York, April 6, 1936.

City of Corning. 1941. Record of Regular Meeting of Honorable Mayor and Members of the Common Council of the City of Corning, New York, July 7, 1941.

City of Corning. 1950. Record of Regular Monthly Meeting of Honorable Mayor and Members of the Common Council of the City of Corning, New York, July 5, 1950.

City of Corning. 1958. Record of Special Meeting of Members of the Common Council of the City of Corning, New York, December 8, 1958.

City of Corning. 1959. Record of Regular Monthly Meeting of Members of the Common Council of the City of Corning, New York, January 5, 1959.

City of Corning. 2018. Utilities Map.

Copp, M.W. 1975. Floods of the Chemung Watershed 1794-1972, "A Day to Remember," June 23, 1972. Marvin W. Copp. 110 pp. (not seen, as cited in STC 2018)

Corning. 2014. Citizen Participation Plan, Study Area Bounded by Pyrex Street, E. Pulteney Street, Post Creek, and the Chemung River, NYSDEC Project ID 851046. Corning Incorporated, Corning, New York. June.

Dalton. 1974. Sanitary Siphon Replacement for the City of Corning, New York. Plans and profiles. Dalton, Dalton, Little, Newport. Cleveland, OH.

Dimitroff, T.P., and L.S. Janes. 1991. *History of the Corning-Painted Post Area: 200 Years in Painted Post Country*. Bookmarks Publishing, London, UK.

EADS. 1979. Susquehanna River Basin, Review of Completed Projects. Reconnaissance Report of Corning-Painted Post, New York, Interior Drainage Facilities. Prepared for Department of

the Army, Baltimore District, Corps of Engineers. Engineering & Associated Design Services, Altoona, PA. April 25.

EDR. 2014. Certified Sanborn® Map Report, Roosevelt St, Corning, NY 14830. Inquiry Number: 3849765.1. Maps provided: 1968, 1948, 1930, 1921. Environmental Data Resources Inc., Milford, CT. February 6.

FEMA. 2002. Flood Insurance Rate Map (FIRM): City of Corning, New York, Community - Panel Number 3607720001E. Federal Emergency Management Agency.

Miller, T.S., J.L. Belli, and R.V. Allen. 1982. Geohydrology of the valley fill aquifer in the Corning area, Steuben County, New York: USGS Open-File Report 82-85, 6 sheets, scale 1:24000. Available at: https://pubs.er.usgs.gov/publication/ofr8285.

NYSDEC. 1973. Memorandum from John O'Connor to File, regarding D.S.R. Work at Corning, date October 2, 1973. New York State Department of Environmental Conservation.

NYSDEC. 1995. New York State Freshwater Wetlands Delineation Manual. July. Available at: http://www.dec.ny.gov/docs/wildlife_pdf/wdelman.pdf.

NYSDEC. 2004. 30 Year Trends in Water Quality of Rivers and Streams in New York State. Available at: https://www.dec.ny.gov/chemical/78979.html.

NYSDEC. 2007. The 2004 Chemung River Basin Waterbody Inventory and Priority Waterbodies List. New York State Department of Environmental Conservation. 163 pp. May.

NYSDEC. 2010. DER-10 / Technical Guidance for Site Investigation and Remediation. DEC Program Policy. New York State Department of Environmental Conservation, Division of Environmental Remediation. Issued May 3, 2010. Errata issued November 7, 2017.

NYSDEC. 2014. Screening and Assessment of Contaminated Sediment. Available at: http://www.dec.ny.gov/docs/fish_marine_pdf/screenasssedfin.pdf. New York State Department of Environmental Conservation, Division of Fish, Wildlife and Marine Resources, Bureau of Habitat. June 24.

NYSDEC. 2017a. Decision Document. Study Area Operable Units (OU) 1, 2 and 5. New York State Department of Environmental Conservation. July 2017. 18 pp.

NYSDEC. 2017b. Decision Document. Study Area Operable Unit (OU) 3. New York State Department of Environmental Conservation. July 2017. 18 pp.

NYSDEC. 2018a. Environmental Resource Mapper. Available at: http://www.dec.ny.gov/animals/38801.html. New York State Department of Environmental Conservation.

NYSDEC. 2018b. New York Nature Explorer. Available at: http://www.dec.ny.gov/natureexplorer/app/location/county/results.2. New York State Department of Environmental Conservation.

Olcot, P.G. 1995. U.S. Geological Survey—Groundwater Atlas of the United States: Segment 12, Connecticut, Maine, Massachusetts, New Hampshire, New York, Rhode Island, Vermont, Hydrologic Investigations Atlas, 730-M.

Rockwood, A.J. 1901. Map Accompanying Report on Corning Dikes. Submitted to E.A. Bond, State Engineer & Surveyor. January 25, 1901.

Sinclair, E.F., and J.S. Spillman. 1997. *The Complete Cut & Engraved Glass of Corning*, Syracuse University Press, Syracuse, NY.

STC. 2018. Local Flood Hazards. Historic Floods in the Southern Tier Central Region of New York. Southern Tier Central Regional Planning & Development Board. Corning, NY. Available at: www.stcplanning.org/usr/Program_Areas/Flood_Mitigation/STC_Historic_Floods.pdf.

USACE. 1941. Record Drawings. Southern New York Flood Control Project, Corning, N.Y. Plans for Corning Flood Protection Project, Section No. 2. U.S. Army Corps of Engineers, U.S. Engineer Office, Binghamton, NY.

USACE. 1956. Chemung River & Post Creek, Additional Drainage Controls, General Plan. Corps of Engineers, U.S. Army, New York District, New York, NY (Maps). January 17.

USACE. 1973. Susquehanna River Flood Control Project Plans for Restoration of Flood Control Project, Corning-Painted Post, N.Y. Prepared by Tippetts, Abbett, McCarthy, Stratton Engineers and Architects of New York, NY for Department of the Army, Baltimore District, Corps of Engineers, Baltimore, MD (Maps).

USACE. 2011. Corning-Painted Post, New York Flood Damage Reduction Systems - Corning-Painted Post Local Flood Protection Project, Revised Final Pre-Inspection Packet for Periodic Inspection No. 01- February 18, 2011.

USDA. 2018. Web Soil Survey. U.S. Department of Agriculture, Natural Resources Conservation Service. Available at: https://websoilsurvey.sc.egov.usda.gov/App/WebSoilSurvey.aspx.

USDOT. 1988. Project Report IV, Draft Design Reports and Supplemental Environmental Impact Statement for Southern Tier Expressway. U.S. Department of Transportation Federal Highway Administration. September 1988.

USFWS. 2018. National Wetlands Inventory Wetlands Mapper. Available at: https://www.fws.gov/wetlands/data/mapper.html. U.S. Fish & Wildlife Service, National Digital Library.

USGS. 2018. USGS 01529950 Chemung River at Corning, NY. Provisional data subject to revision. Available at: https://waterdata.usgs.gov/nwis/uv?site_no=01529950 U.S. Geological Survey, National Water Information System.

Weston. 2014a. 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. Prepared for Corning Incorporated. Weston Solutions, Inc., West Chester, PA. 81 pp. June.

Weston. 2014b. Study Area Characterization Work Plan, Addendum Number 1, Additional Field Investigation Activities. Prepared for Corning Incorporated. Weston Solutions, Inc. 15 pp. October 24.

Weston. 2015a. Study Area Characterization Work Plan Addendum Number 2, Additional Field Investigation Activities. Prepared for Corning Incorporated. Weston Solutions, Inc. 23 pp. May 11.

Weston. 2015b. Study Area Characterization Work Plan Addendum Number 3, Additional Field Investigation Activities. Prepared for Corning Incorporated. Weston Solutions, Inc. 15 pp. March 20.

Weston. 2016a. Interim Remedial Measures Work Plan, Corning-Painted Post School District Property. Prepared for Corning Incorporated. Weston Solutions, Inc. 161 pp. November 10.

Weston. 2016b. Interim Remedial Measures Work Plan, City of Corning Memorial Stadium. Prepared for Corning Incorporated. Weston Solutions, Inc. 175 pp. November 10.

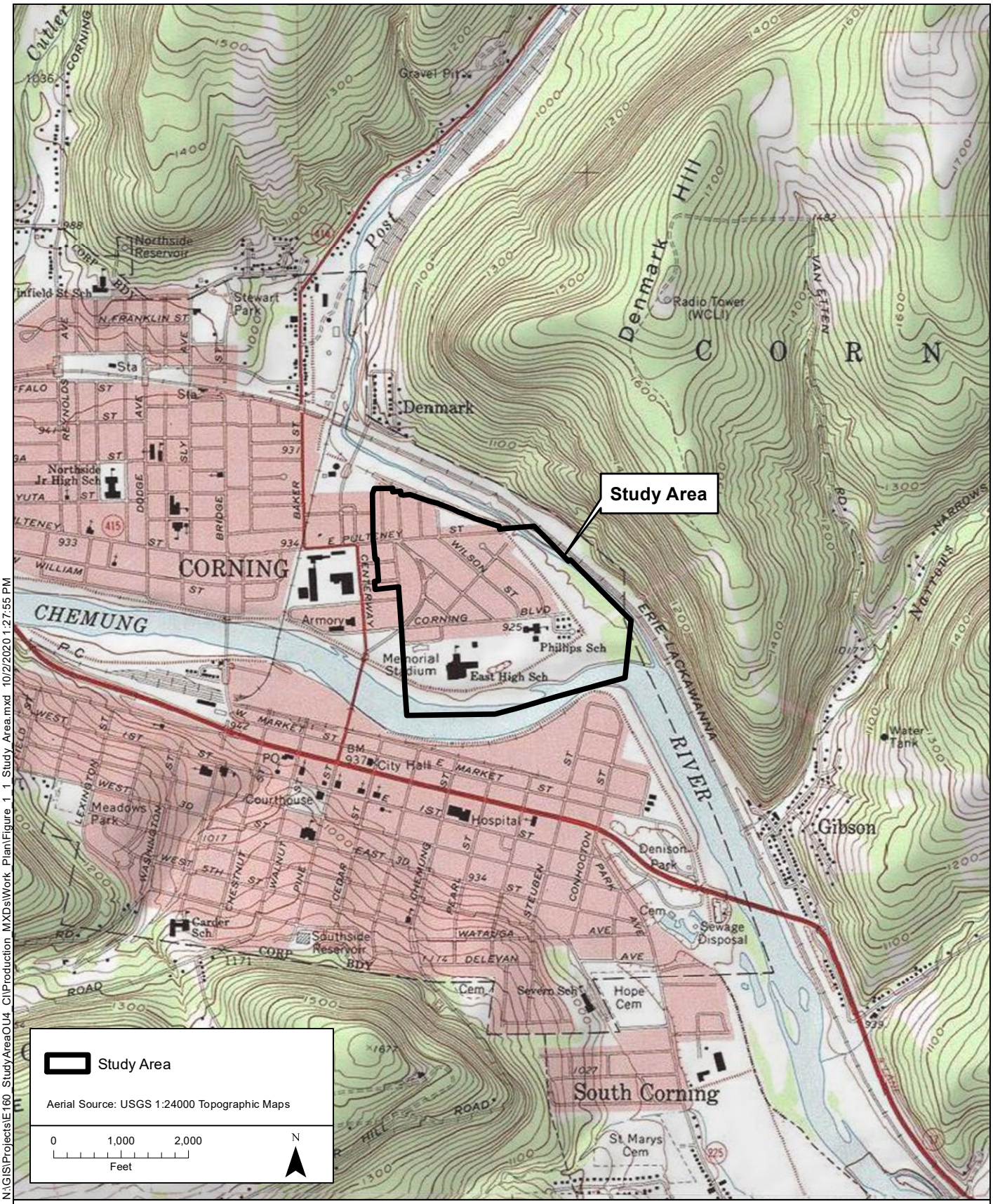
Weston. 2016c. Interim Remedial Measures Work Plan, Corning Christian Academy Property. Prepared for Corning Incorporated. Weston Solutions, Inc. 161 pp. November 10.

Weston. 2017a. Study Area Characterization Work Plan Addendum Number 4, Additional Field Investigation Activities. Prepared for Corning Incorporated. Weston Solutions, Inc. 11 pp. March 10.

Weston. 2017b. Focused Feasibility Study / Alternatives Analysis, Residential Areas (OU1, OU2, and OU5). Prepared for Corning Incorporated. Weston Solutions, Inc. 95 pp. March 23.

Weston. 2018. Remedial Action Work Plan, Residential Areas (OU1, OU2 and OU5), Study Area, Corning, NY. NYSDEC Project ID 851046. Prepared for Corning Incorporated. Weston Solutions, Inc., 132 pp. April 6.

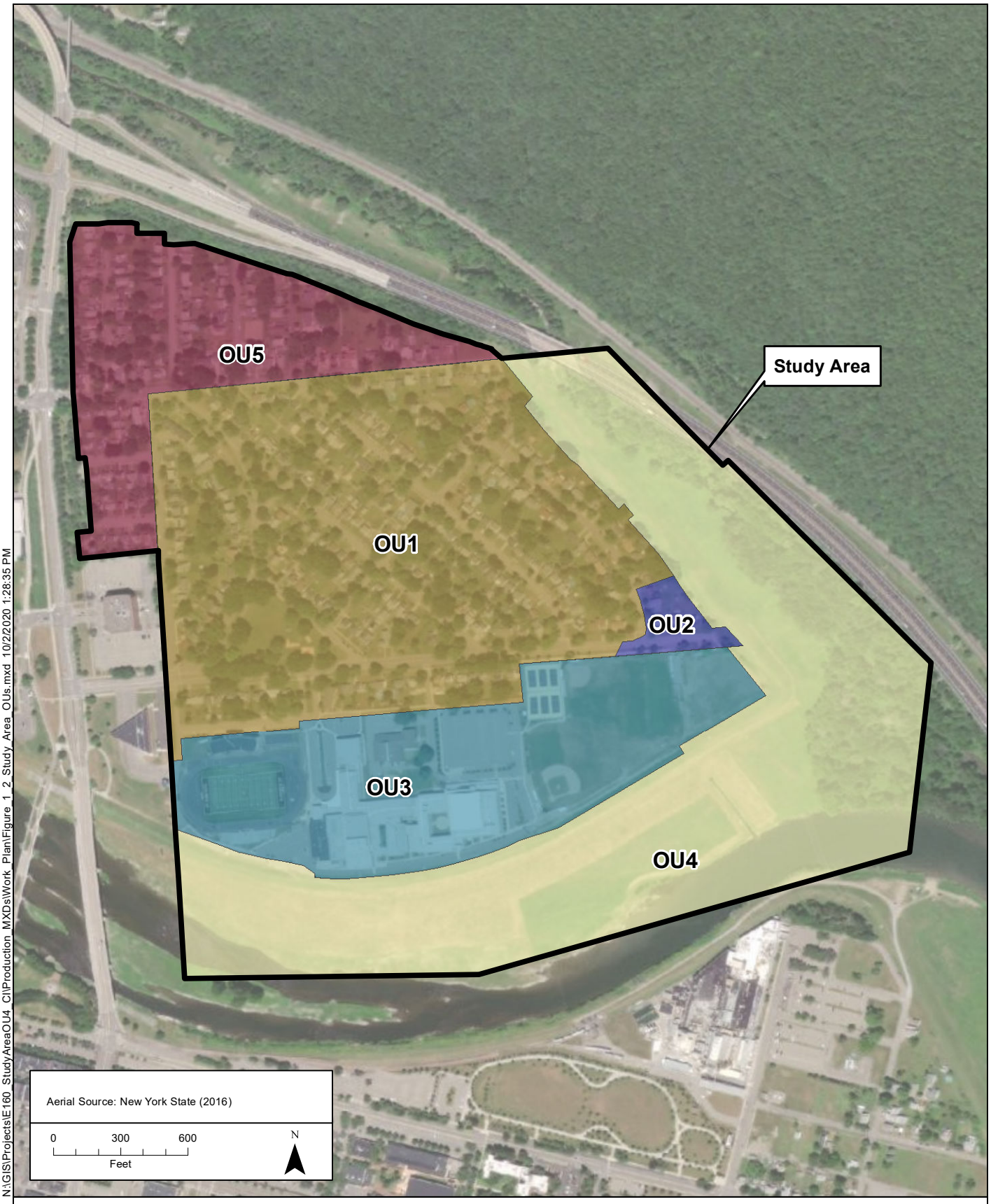
FIGURES



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_1_1_Study_Area.mxd 10/2/2020 1:27:55 PM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

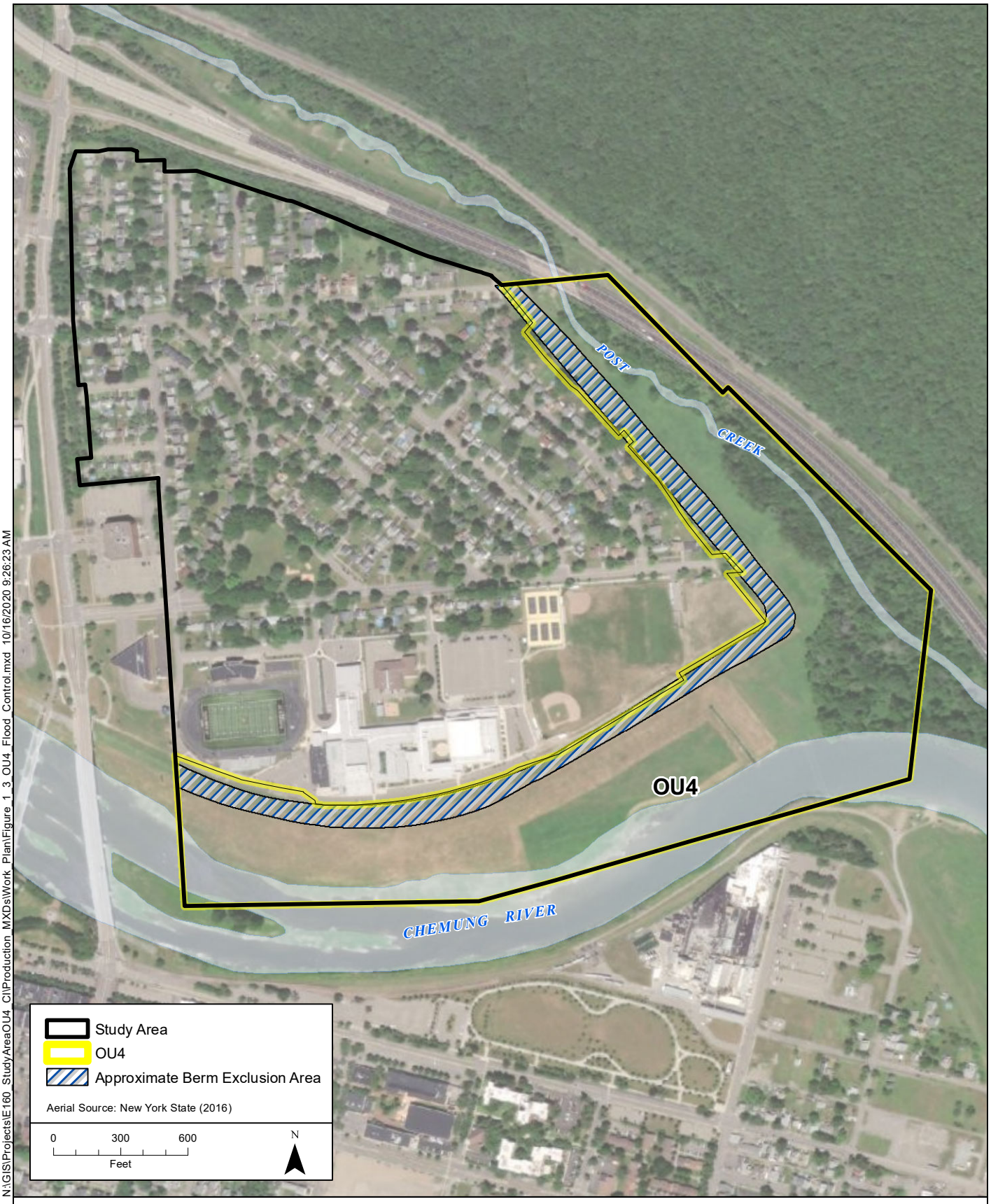
Figure 1-1.
Location of Study Area
Study Area OU4 Site Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_1_2_Study_Area_OUs.mxd 10/2/2020 1:28:35 PM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

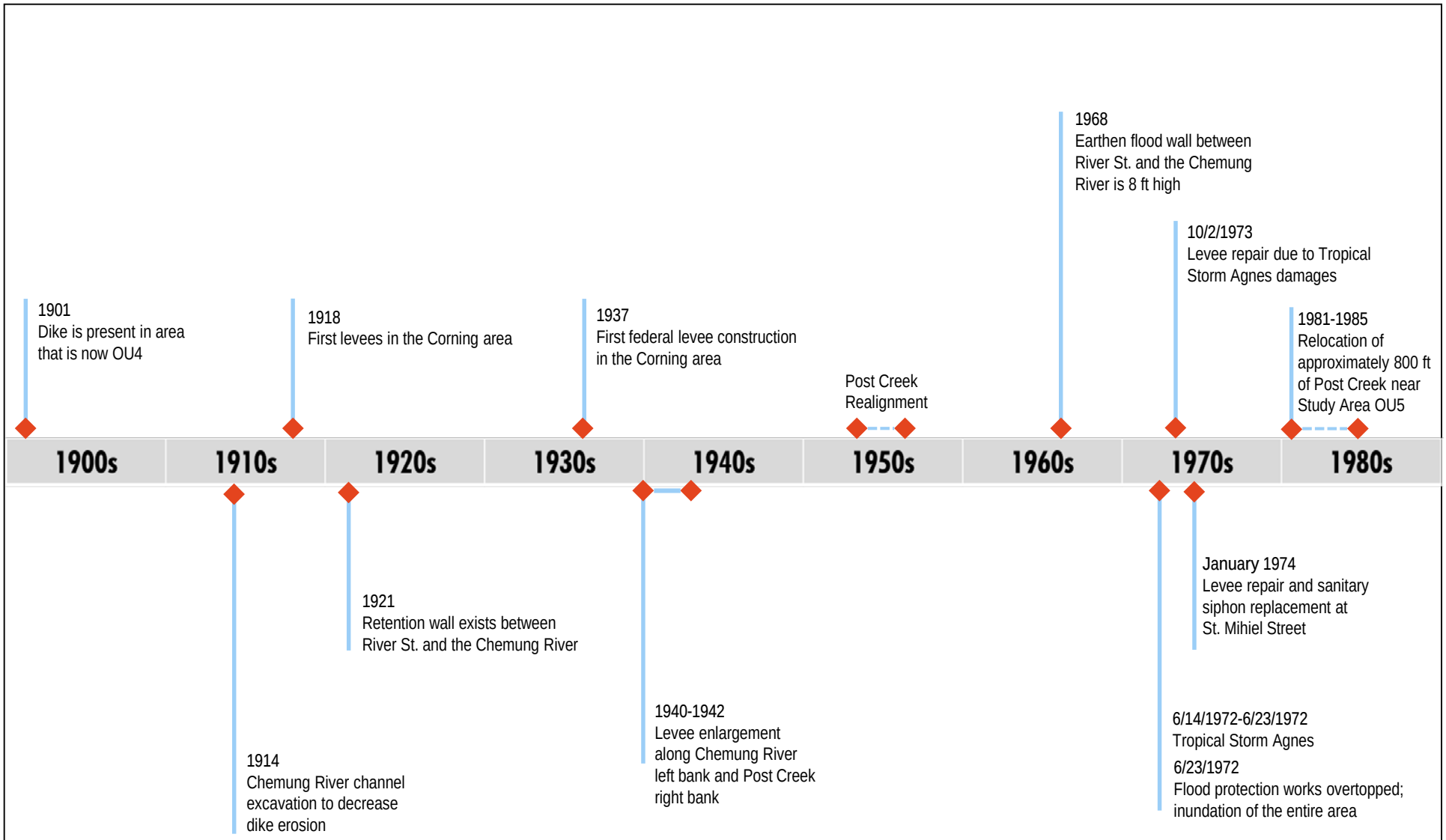
Figure 1-2.
Study Area Operable Units
Study Area OU4 Site Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_1_3_OU4_Flood_Control.mxd 10/16/2020 9:26:23 AM

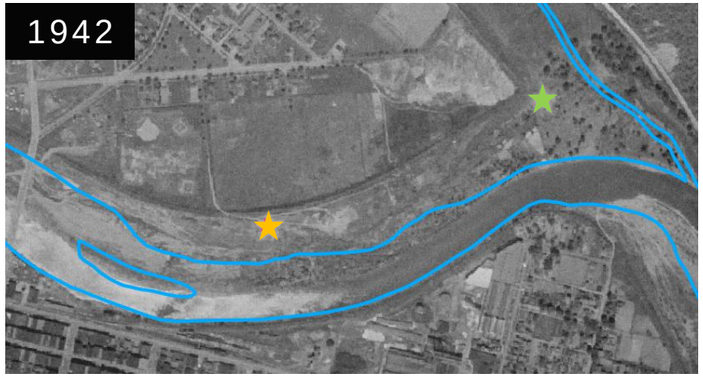
integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

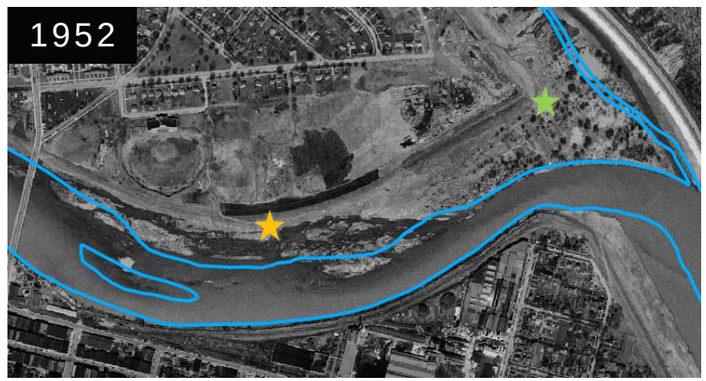
Figure 1-3.
OU4 Flood Control Areas
Study Area OU4 Site Characterization Work Plan

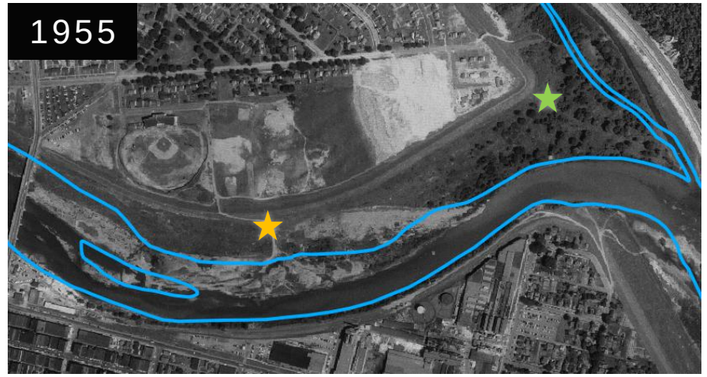


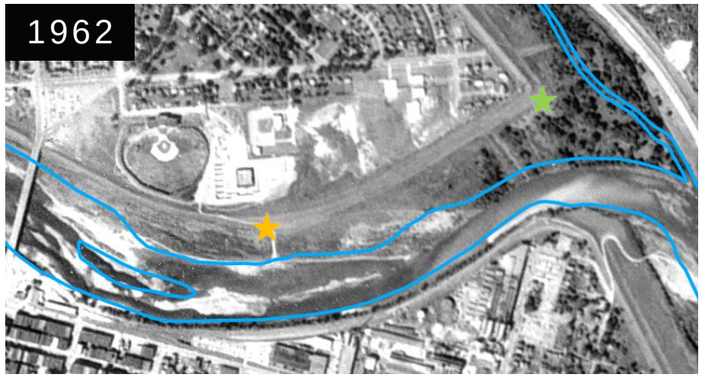
integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

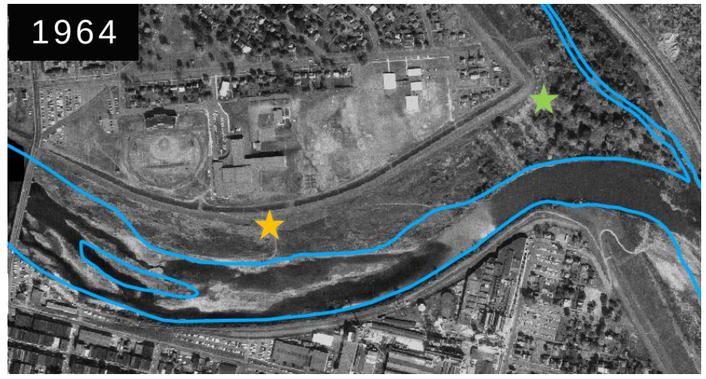
Figure 2-1.
Summary of OU4 Construction Activities
Study Area OU4 Characterization Work Plan

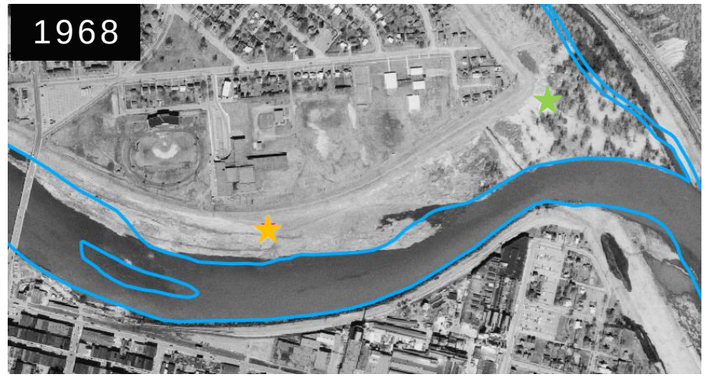


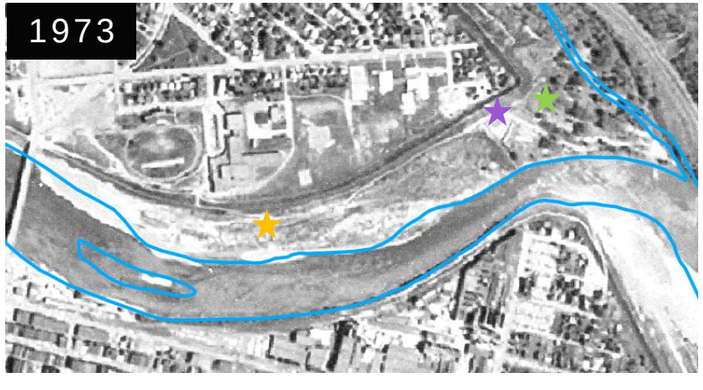








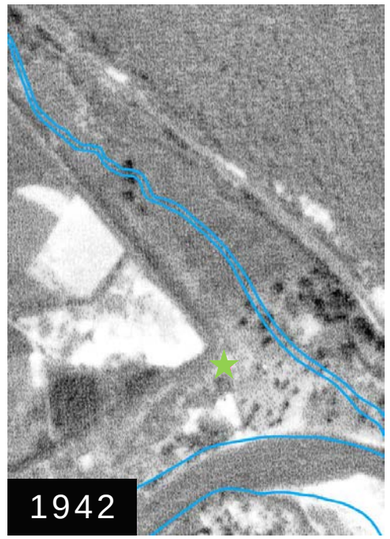


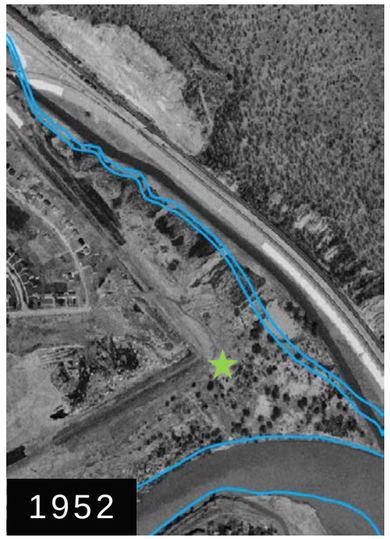


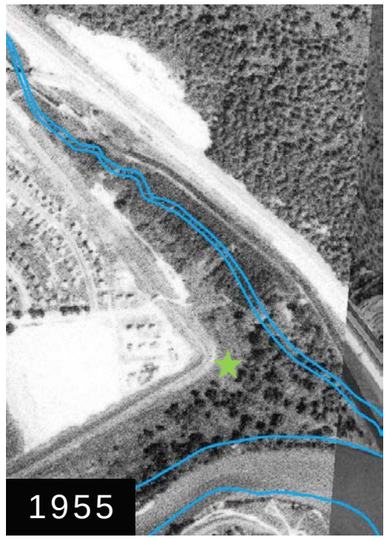
integral engineering pc.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

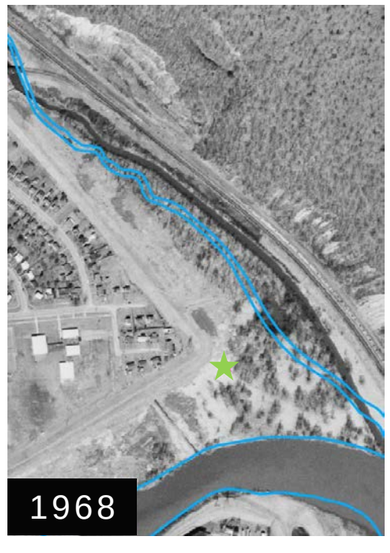
Notes:
The blue line denotes the present day location of Post Creek and the Chemung River.
The green star indicates an area where vegetative cover transitions from low-growing vegetation to larger plants.
The orange star indicates the location of a road.
The purple star indicates berm modifications following Tropical Storm Agnes.

Figure 2-2.
OU4 Chemung River Photograph Chronology, 1942 – 2012
Study Area OU4 Characterization Work Plan





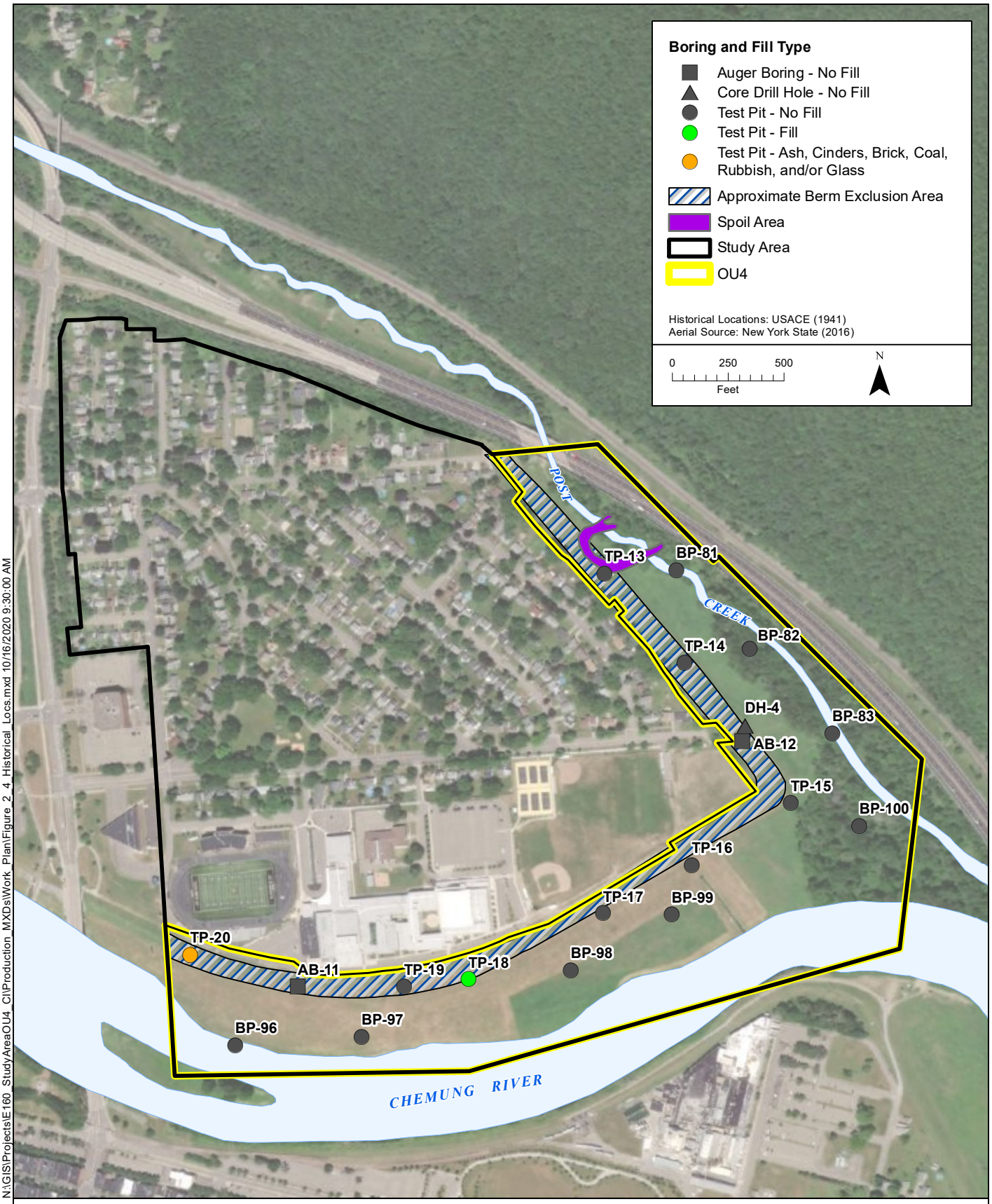




integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Notes:
The blue line denotes the present day location of Post Creek and the Chemung River.
The green star indicates an area where vegetative cover transitions from low-growing vegetation to larger plants.

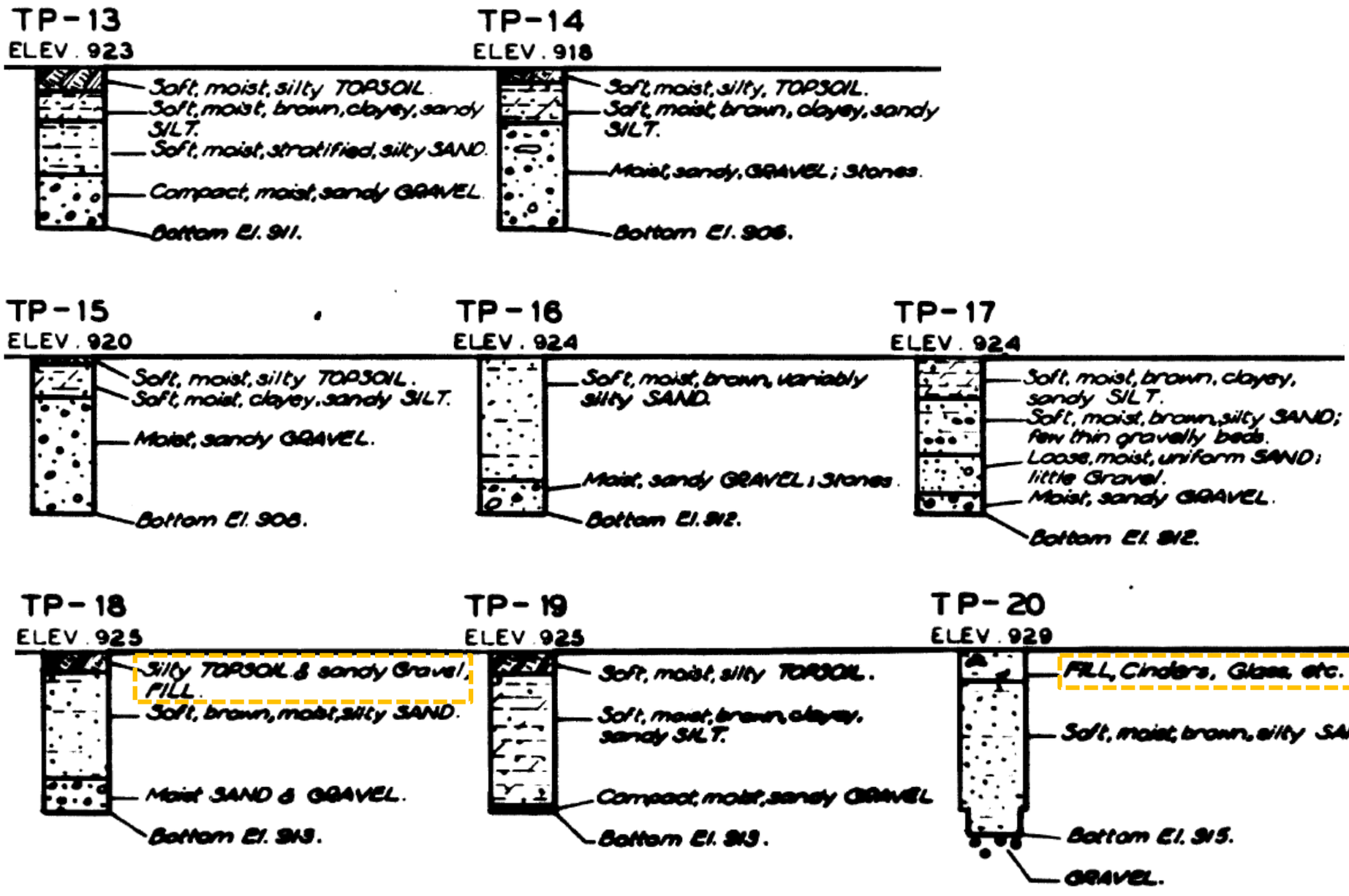
Figure 2-3.
OU4 Post Creek Photograph Chronology, 1942 – 2012
Study Area OU4 Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_2_4_Historical_Locs.mxd 10/16/2020 9:30:00 AM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Figure 2-4.
Historical Soil Boring and Test Pit Locations
Study Area OU4 Site Characterization Work Plan

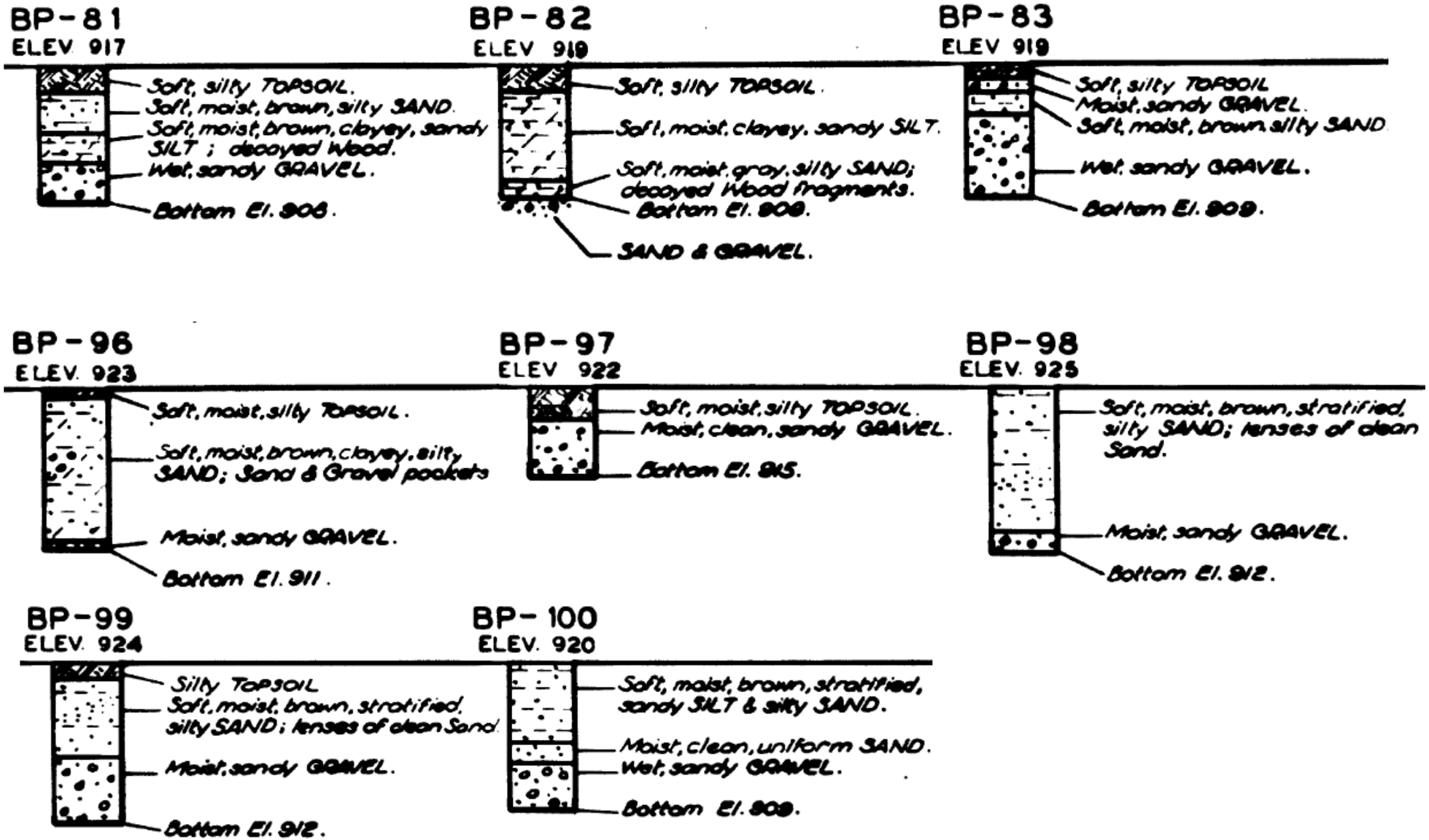


integral engineering pc.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Notes:
Soil log images from USACE (1941).

The dashed orange line indicates fill material containing glass.

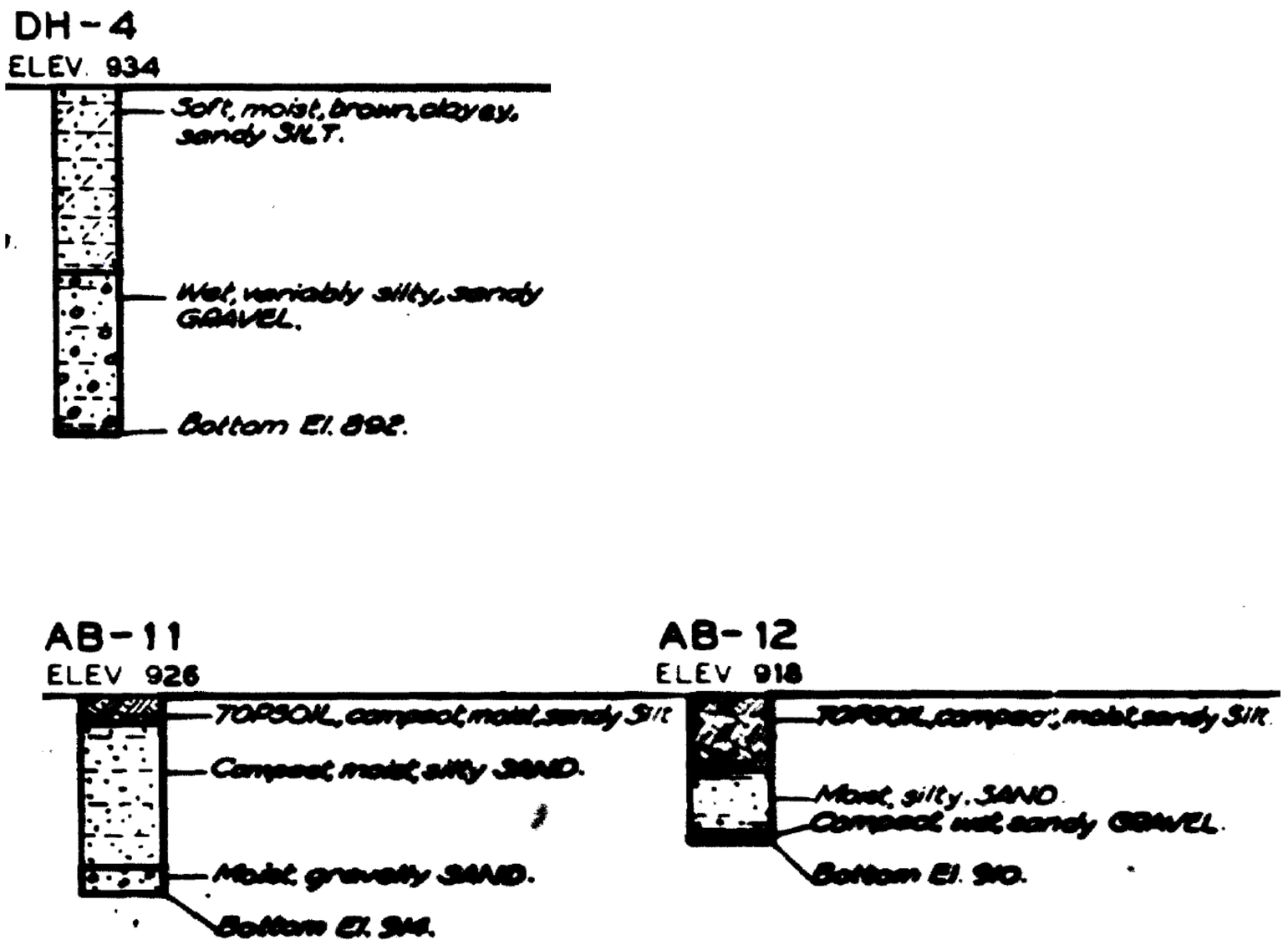
Figure 2-5a.
OU4 Historical Soil Logs – Test Pits
Study Area OU4 Characterization Work Plan



integral engineering pc
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Notes:
Core images from USACE (1941).

Figure 2-5b.
OU4 Historical Soil Borings – Borrow Test Pits
Study Area OU4 Characterization Work Plan



integral engineering pc.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Notes:
Core images from USACE (1941).

Figure 2-5c.
OU4 Historical Soil Borings – Drill and Auger Holes
Study Area OU4 Characterization Work Plan

Grassy vegetation growing on flood control dike and floodplain in OU4; looking to the north from the southern berm.

Herbaceous and woody vegetation growing in OU4 near Post Creek.

Grassy vegetation growing on flood control dike and floodplain in OU4; looking to the southwest and Chemung River from the southern berm.

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

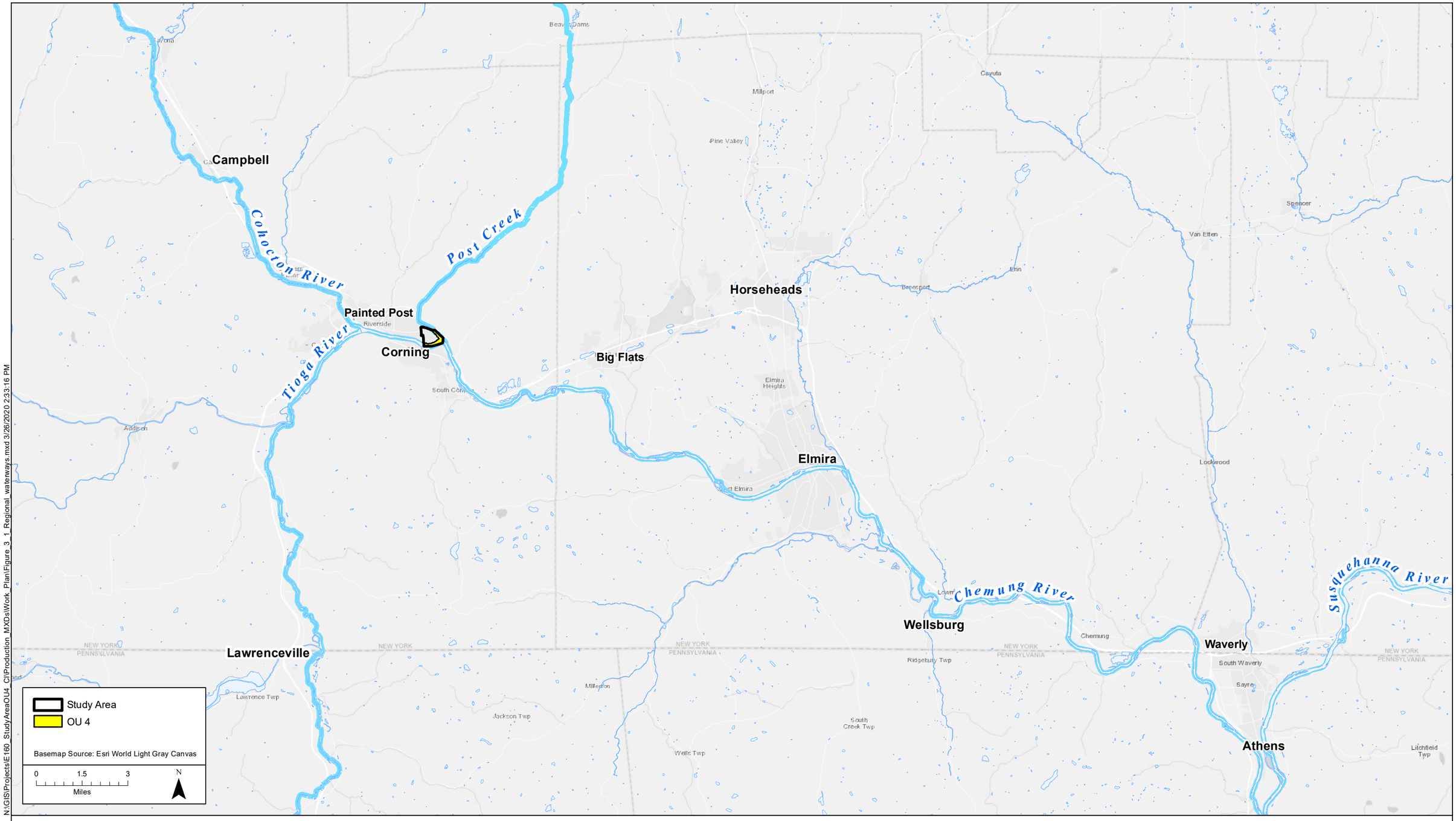
Figure 2-6.
OU4 Photographs of Floodplain Vegetation, May 2018
Study Area OU4 Characterization Work Plan

Mixed vegetation and exposed banks in OU4 along Post Creek.

Grassy vegetation and exposed bank in OU4 along the Chemung River looking west.

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

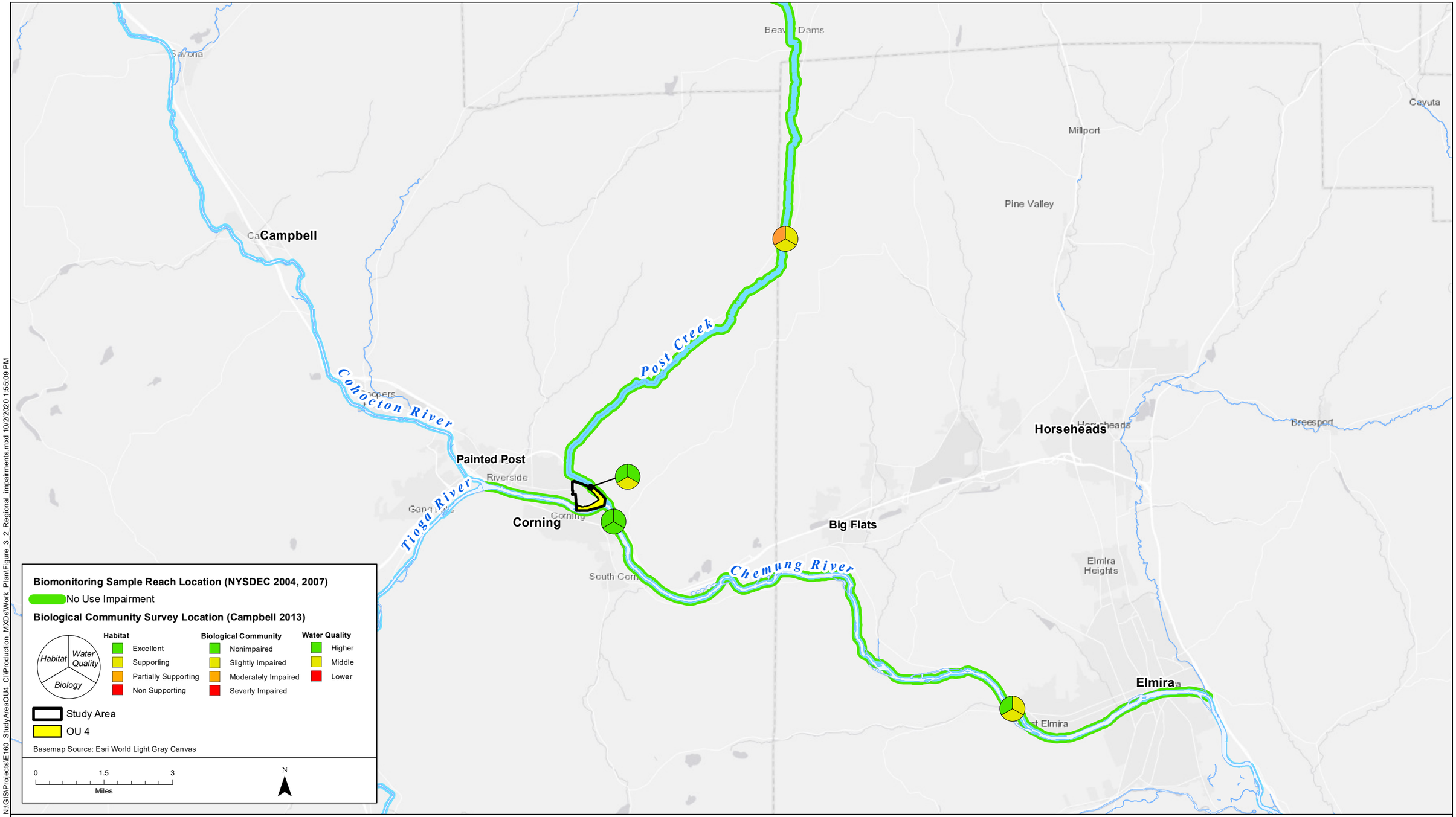
Figure 2-7.
OU4 Photographs of Unvegetated River Banks, May 2018
Study Area OU4 Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_3_1_Regional_waterways.mxd 3/26/2020 2:33:16 PM

integral engineering pc.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

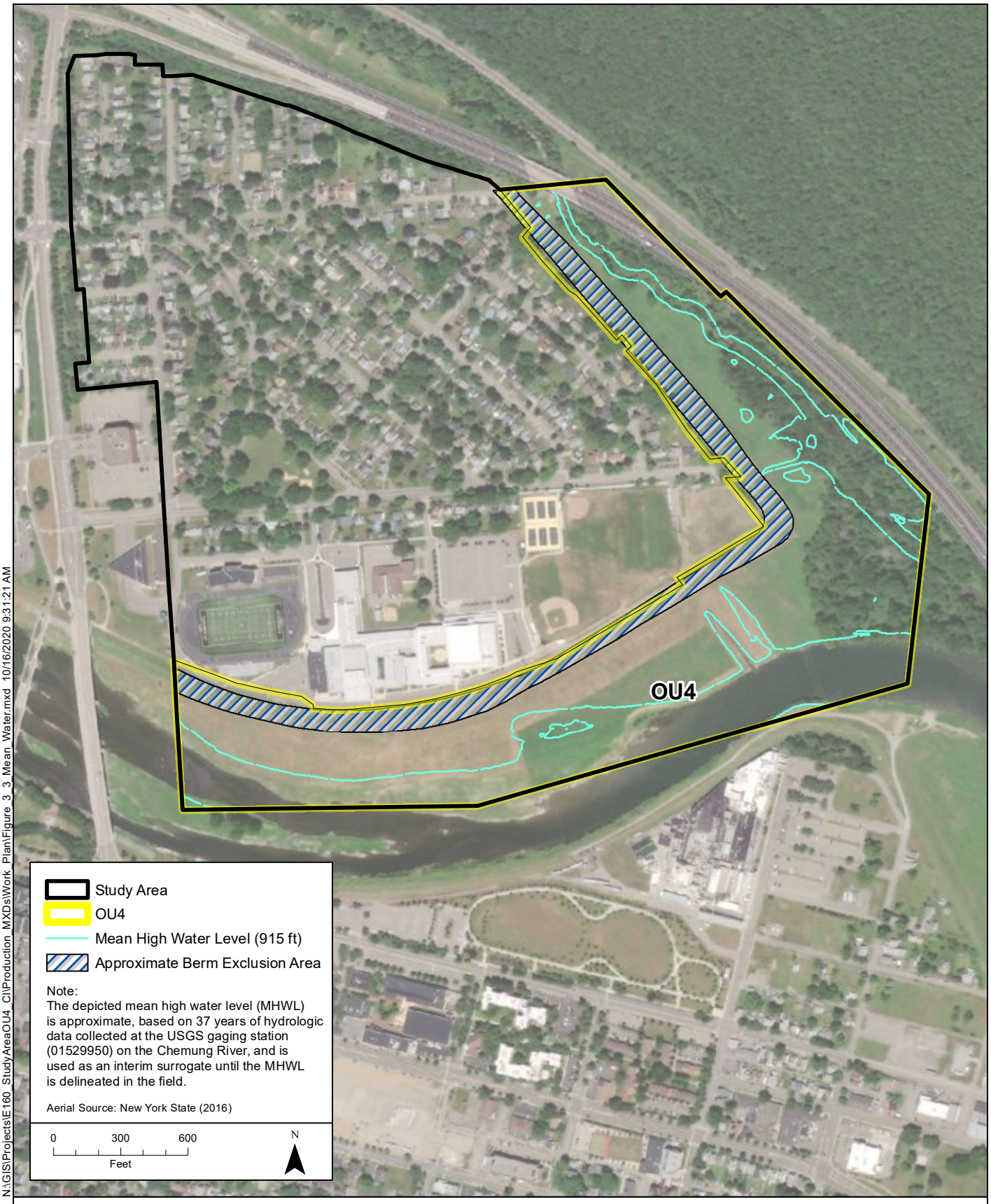
Figure 3-1.
Major Regional Waterways
Study Area OU4 Site Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_3_2_Regional_impairments.mxd 10/2/2020 1:55:09 PM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

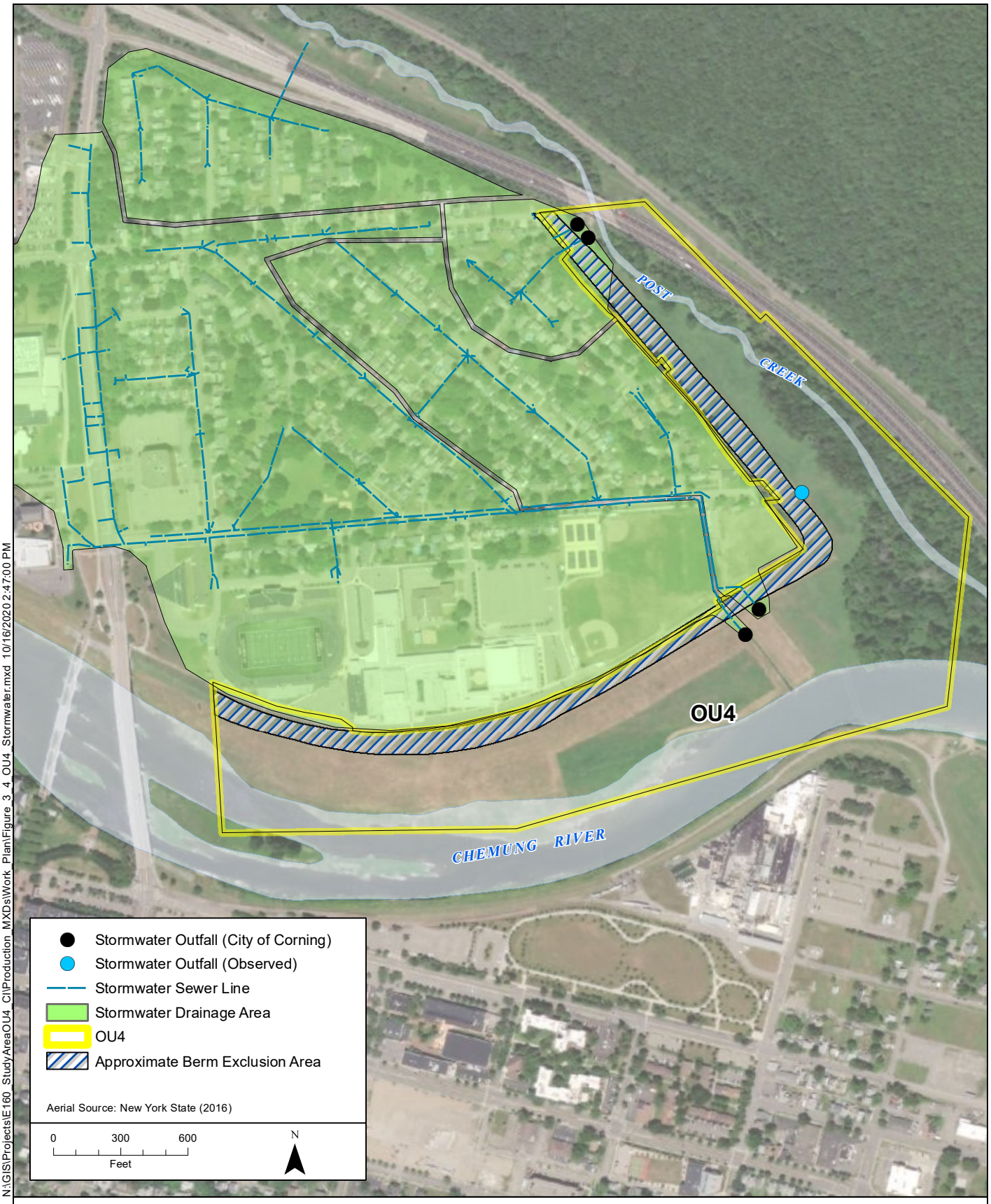
Figure 3-2.
Regional Water Quality Studies
Study Area OU4 Site Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_3_3_Mean_Water.mxd 10/16/2020 9:31:21 AM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

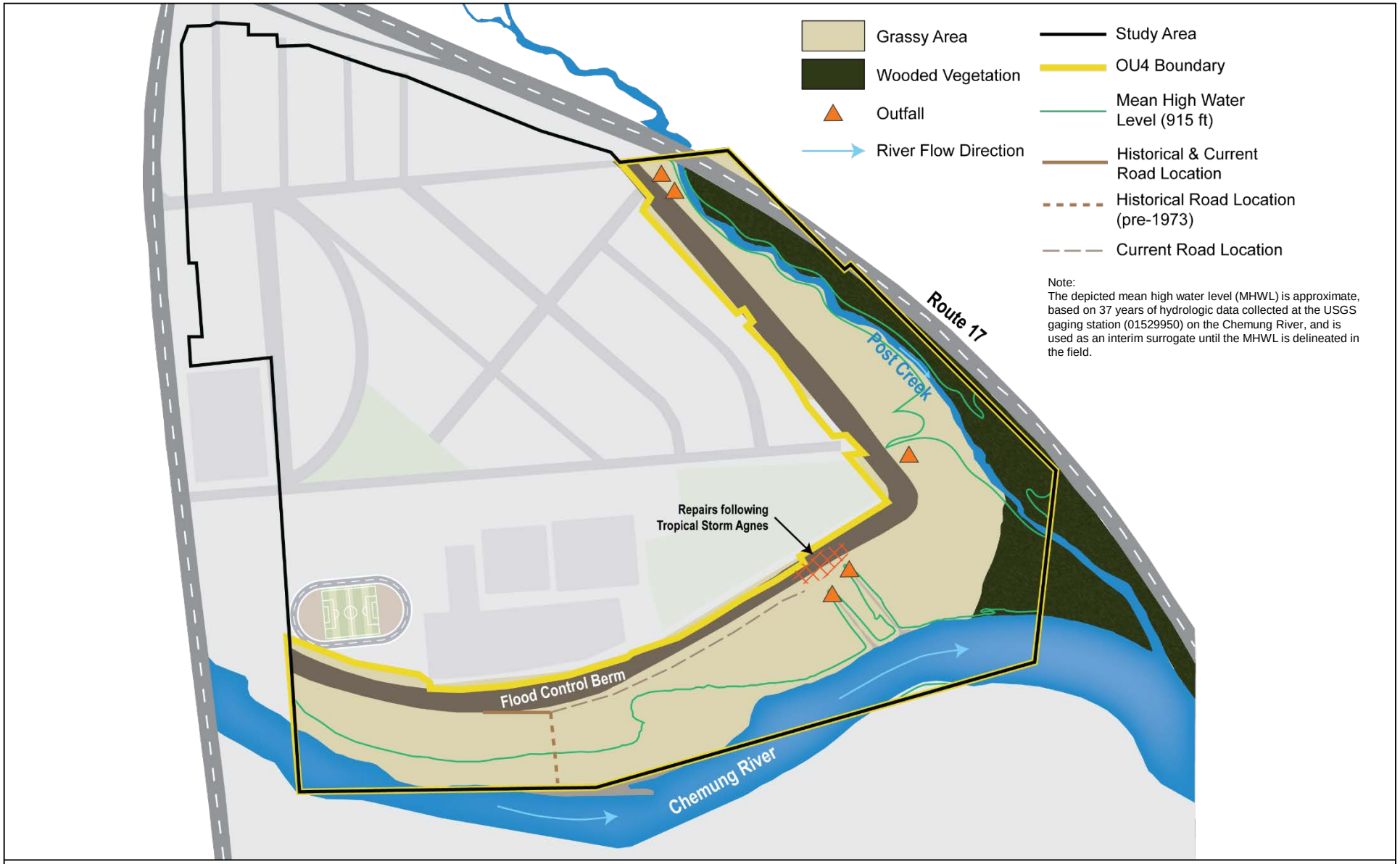
Figure 3-3.
Mean High Water Level, OU4 Flood Control Areas
Study Area OU4 Site Characterization Work Plan



N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_3_4_OU4_Stormwater.mxd 10/16/2020 2:47:00 PM

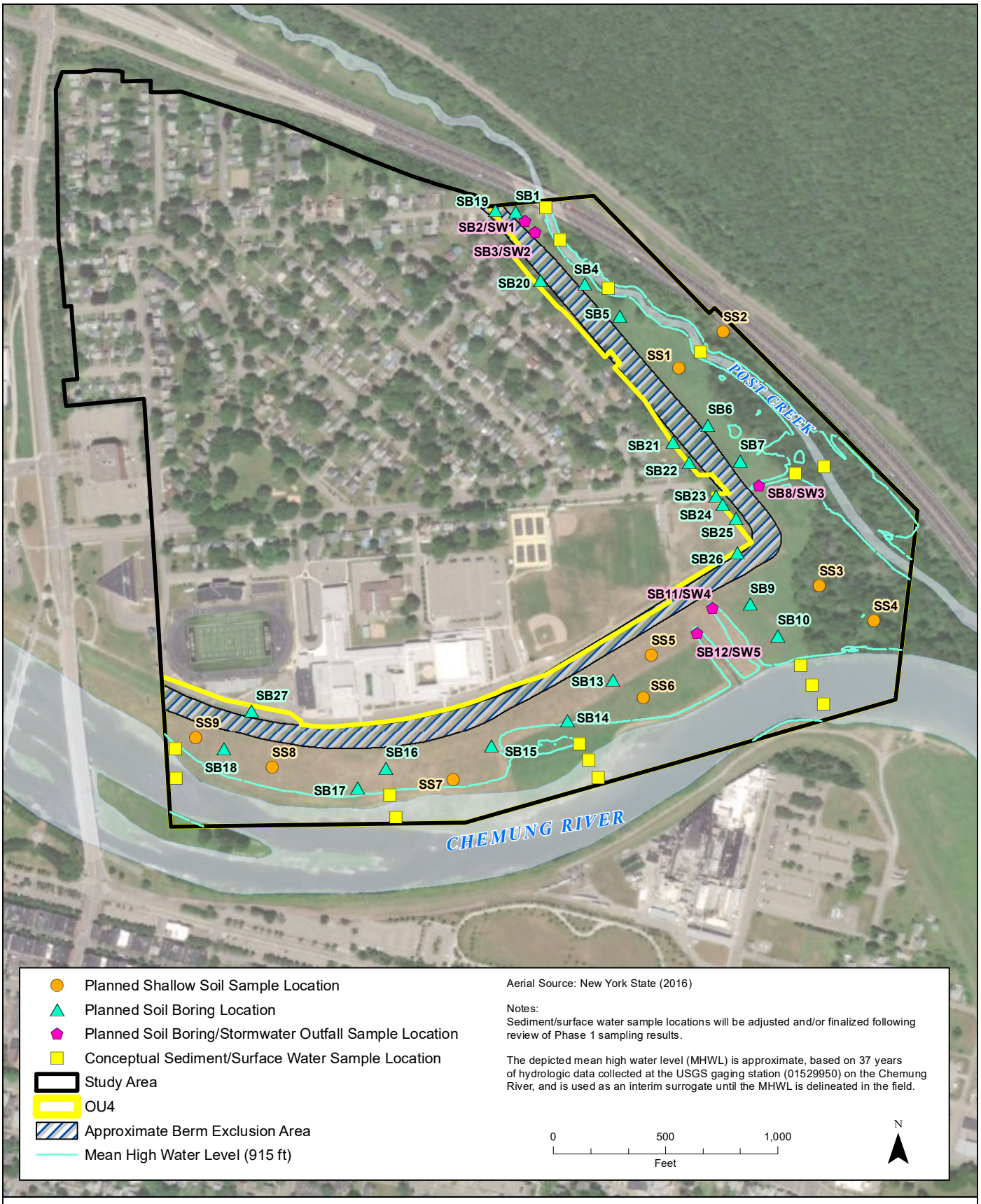
integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Figure 3-4.
Study Area and OU4 Storm Drainage
Study Area OU4 Site Characterization Work Plan



integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Figure 4-1.
OU4 Features
Study Area OU4 Characterization Work Plan

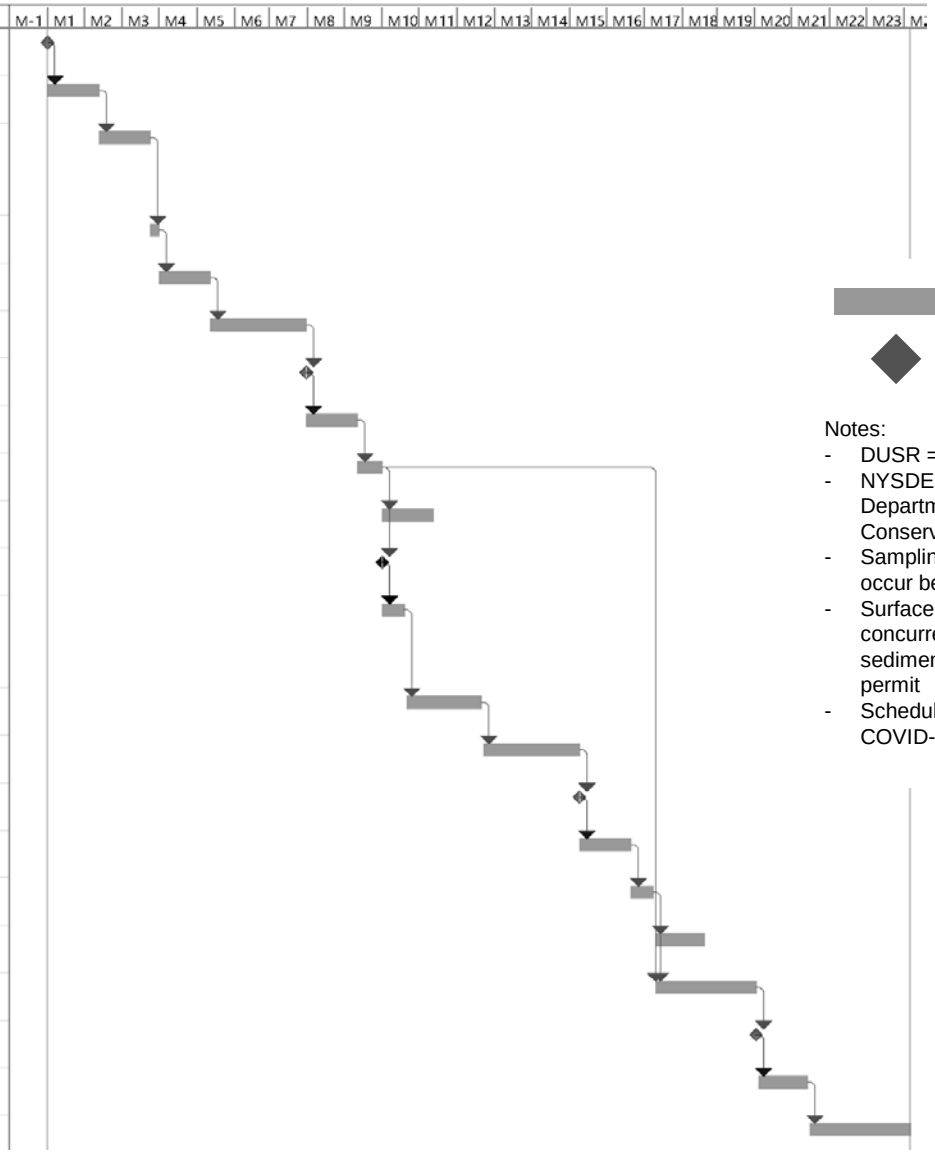


N:\GIS\Projects\E160_StudyAreaOU4_CI\Production_MXDs\Work_Plan\Figure_5_1_OU4_sample_locs.mxd 10/16/2020 3:07:17 PM

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Figure 5-1.
Sampling Locations
Study Area OU4 Site Characterization Work Plan

Task Name	Duration
NYSDEC Approval	0 days
Access Agreement and Mobilization	30 days
Boundary, Wetlands, and Biological Surveys	30 days
Utility Clearances	5 days
Soil Boring & Surface Soil Sampling	30 days
Sample Analysis & Validation	56 days
Draft DUSR to NYSDEC	0 days
NYSDEC Review DUSR	30 days
Finalize DUSR	14 days
Submittal of Data to Property Owners	30 days
Sediment Sampling Locations to NYSDEC	0 days
NYSDEC Review/Approval of Sediment Sampling Locations	14 days
Sediment Sampling	45 days
Sample Analysis & Validation	56 days
Draft DUSR to NYSDEC	0 days
NYSDEC Review DUSR	30 days
Finalize DUSR	14 days
Submittal of Data to Property Owners	30 days
Prepare Draft Characterization Report	60 days
Draft to NYSDEC	0 days
NYSDEC Comments	30 days
Prepare Final Characterization Report	60 days



Notes:
- DUSR = data usability summary report
- NYSDEC = New York State Department of Environmental Conservation
- Sampling activities are anticipated to occur between June and September
- Surface water sampling will occur concurrently with either the soil or sediment sampling, as conditions permit
- Schedule may be impacted due to COVID-19-related protocols or delays

integral engineering p.c.
1001 6th Ave, 11th Floor
New York, NY 10018
www.integral-corp.com

Figure 6-1.
OU4 Characterization Schedule
Study Area OU4 Characterization Work Plan

TABLES

Table 2-1. Historical Exploration Logs in OU4 Flood Control Areas

Station ID	Core Top Elevation (ft)	Core Bottom Elevation (ft)	Core Depth (ft)	Station Type	Fill Type	Fill Layer	Fill Upper Depth (ft)	Fill Lower Depth (ft)	Fill Description
DH-4	934	892	42	Core drill hole	No fill	--	--	--	--
AB-11	926	914	12	Auger boring	No fill	--	--	--	--
AB-12	918	910	8	Auger boring	No fill	--	--	--	--
TP-13	923	911	12	Test pit	No fill	--	--	--	--
TP-14	918	906	12	Test pit	No fill	--	--	--	--
TP-15	920	908	12	Test pit	No fill	--	--	--	--
TP-16	924	912	12	Test pit	No fill	--	--	--	--
TP-17	924	912	12	Test pit	No fill	--	--	--	--
TP-18	925	913	12	Test pit	Plain soil or wood	Surface	0	2	Silty topsoil and sandy gravel, fill
TP-19	925	913	12	Test pit	No fill	--	--	--	--
TP-20	929	915	14	Test pit	Cinders, glass	Surface	0	3	Fill. Cinders, glass, etc.
BP-81	917	906	11	Borrow test pit	No fill	--	--	--	--
BP-82	919	909	10	Borrow test pit	No fill	--	--	--	--
BP-83	919	909	10	Borrow test pit	No fill	--	--	--	--
BP-96	923	911	12	Borrow test pit	No fill	--	--	--	--
BP-97	922	915	7	Borrow test pit	No fill	--	--	--	--
BP-98	925	912	13	Borrow test pit	No fill	--	--	--	--
BP-99	924	912	12	Borrow test pit	No fill	--	--	--	--
BP-100	920	909	11	Borrow test pit	No fill	--	--	--	--

Source: USACE (1941)

Table 5-1. Phase 1 Sample Locations

Sample ID	Sample Location Rationale	Sample Type[a]	Target Coordinates	
			Longitude	Latitude
SB1	Observed ash, brick, and/or glass	Soil Boring	-77.04540	42.15202
SB2/SW1	Storm water outfall	Soil Boring/SW	-77.04524	42.15191
SB3/SW2	Storm water outfall	Soil Boring/SW	-77.04508	42.15178
SB4	Spoil Area	Soil Boring	-77.04426	42.15115
SB5	Spoil Area	Soil Boring	-77.04367	42.15075
SB6	Potential disturbance area	Soil Boring	-77.04221	42.14942
SB7	Adjacent to observed ash, brick, and/or glass	Soil Boring	-77.04168	42.14898
SB8/SW3	Storm water outfall	Soil Boring/SW	-77.04137	42.14869
SB9	Berm breach area	Soil Boring	-77.04150	42.14724
SB10	Berm breach area	Soil Boring	-77.04104	42.14684
SB11/SW4	Storm water outfall	Soil Boring/SW	-77.04212	42.14718
SB12/SW5	Storm water outfall	Soil Boring/SW	-77.04237	42.14688
SB13	Spatial Coverage	Soil Boring	-77.04375	42.14629
SB14	Confirmation of historic data	Soil Boring	-77.04450	42.14579
SB15	Disturbance - Former road location	Soil Boring	-77.04575	42.14547
SB16	Disturbance - Former road location	Soil Boring	-77.04748	42.14520
SB17	Confirmation of historic data	Soil Boring	-77.04794	42.14495
SB18	Spatial Coverage	Soil Boring	-77.05015	42.14542
SB19	Spatial Coverage	Soil Boring	-77.04573	42.15203
SB20	Spatial Coverage	Soil Boring	-77.04498	42.15118
SB21	Observed ash, brick, and/or glass	Soil Boring	-77.04279	42.14921
SB22	Observed ash, brick, and/or glass	Soil Boring	-77.04252	42.14896
SB23	Observed ash, brick, and/or glass	Soil Boring	-77.04207	42.14855
SB24	Observed ash, brick, and/or glass	Soil Boring	-77.04197	42.14845
SB25	Observed ash, brick, and/or glass	Soil Boring	-77.04174	42.14828
SB26	Observed ash, brick, and/or glass	Soil Boring	-77.04172	42.14786
SB27	Spatial Coverage	Soil Boring	-77.04970	42.14589
SS1	Spatial coverage	Shallow Soil	-77.04269	42.15012
SS2	Spatial coverage	Shallow Soil	-77.04197	42.15057
SS3	Spatial coverage	Shallow Soil	-77.04036	42.14746
SS4	Spatial coverage	Shallow Soil	-77.03946	42.14704
SS5	Spatial coverage	Shallow Soil	-77.04312	42.14660
SS6	Spatial coverage	Shallow Soil	-77.04325	42.14607
SS7	Spatial coverage	Shallow Soil	-77.04637	42.14506
SS8	Spatial coverage	Shallow Soil	-77.04935	42.14520
SS9	Spatial coverage	Shallow Soil	-77.05061	42.14556

Notes:
Samples will undergo analyses listed in QAPP Table C2-2 (Appendix C).
Sediment sample locations will be established following completion of Phase 1 sampling.

[a] SW = surface water

APPENDIX A

HEALTH AND SAFETY PLAN

CHARACTERIZATION WORK PLAN:
STUDY AREA OPERABLE UNIT 4 FLOOD CONTROL AREAS

NYSDEC Project ID 851046, Corning, New York
Health and Safety Plan

Prepared for
Corning Incorporated
Corning, NY

Prepared by

1001 6th Avenue
11th Floor
New York, NY 10018

November 12, 2020

Affiliated with Integral Consulting Inc.

CONTENTS

LIST OF ATTACHMENTS

ACRONYMS AND ABBREVIATIONS

CFR	Code of Federal Regulations
CHSM	corporate health and safety manager
COC	constituent of concern
CPR	cardiopulmonary resuscitation
HASP	health and safety plan
IDLH	immediately dangerous to life and health
Integral	Integral Engineering, P.C.
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration
OU	operable unit
PEL	permissible exposure limit
PID	photoionization detector
PPE	personal protective equipment
SSO	Study Area OU4 safety officer
STEL	short-term exposure limit
VOC	volatile organic compound
Work Plan	Study Area OU4 Characterization Work Plan

HEALTH AND SAFETY PLAN APPROVAL

This health and safety plan has been reviewed and approved for soil sampling in Study Area OU4. OU4 includes areas along the eastern and southern boundaries of the Study Area, consisting of the earthen berm and lands between the eastern boundary of OU1, OU2, OU3, and Post Creek and Highway 17/Interstate 86 to the east, as well as the berm and lands between the southern boundary of OU3 and the Chemung River in the City of Corning, New York.

Project Manager	Date
Corporate Health and Safety Manager	Date

HEALTH AND SAFETY PLAN ACKNOWLEDGMENT

In the absence of an appropriate subcontractor or consultant health and safety plan, and with the written approval of Integral Engineering, P.C. (Integral) corporate health and safety manager, the subcontractor or consultant may utilize the Integral health and safety plan (HASP), provided there is written concurrence from the subcontractor or consultant that they will directly administer the plan for their employees and assume all risks associated with any possible errors or omissions in the plan. This HASP does not cover any construction activities. The Integral HASP is a minimum standard for Study Area OU4 and will be strictly enforced for all Integral personnel, or its subcontractors or consultants where applicable.

I have reviewed the HASP prepared by Integral, dated November 12, 2020, for the Study Area OU4 fieldwork. I understand the purpose of the plan, and I consent to adhere to its policies, procedures, and guidelines while an employee of Integral, or its subcontractors or consultants. I have had an opportunity to ask questions regarding this plan, which have been answered satisfactorily by Integral.

Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date

1 INTRODUCTION

It is the policy of Integral Engineering, P.C. (Integral) to provide a safe and healthful work environment that is compliant with applicable regulations. No aspect of the work is more important than protecting the health and safety of all workers.

This health and safety plan (HASP) provides general health and safety provisions to protect workers from potential hazards during field activities at the Study Area Operable Unit (OU) 4 located in Corning, New York. This HASP has been prepared in accordance with federal Occupational Safety and Health Administration (OSHA) safety regulations (29 CFR [Code of Federal Regulations] 1910 and 29 CFR 1926).

Attachments to the HASP provide a Study Area OU4-specific map and specific routes to the hospital from the Study Area (Attachment 1), regulatory notices (Attachment 2), safety procedures (Attachment 3), safety data sheets (Attachment 4), an employee exposure/injury incident report (Attachment 5), a near-miss incident report (Attachment 6), and COVID-19 site and preventative measures plans (Attachment 7).

This HASP has been prepared to identify potential OU4 hazards to the extent possible based on information available to Integral. Integral cannot guarantee the health or safety of any person entering OU4. Strict adherence to the health and safety guidelines set forth herein will reduce the potential for injury and illness at OU4. The health and safety guidelines in this plan were prepared specifically for OU4 and should not be used elsewhere without prior evaluation by trained health and safety personnel.

A copy of this HASP must be in the custody of the field crew during field activities. All individuals performing fieldwork must read, understand, and comply with this plan before undertaking field activities. Once the information has been read and understood, the individual must sign the Health and Safety Plan Acknowledgment form provided as part of this plan. The signed form will become part of the project file.

This plan may be modified at any time based on the judgment of the Integral Study Area OU4 safety officer (SSO) in consultation with the project manager and Integral corporate health and safety manager (CHSM) or designee. Any modification will be presented to the OU4 team during a safety briefing and will be recorded in the field logbook.

1.1 OBJECTIVES AND METHODS

Field activities referenced in this HASP are associated with the characterization of soil, sediment, and surface water at the Study Area OU4, in the City of Corning, New York. This HASP outlines the health and safety considerations for field activities at OU4.

Study Area OU4 Characterization Work Plan (Work Plan) is the master document, which describes characterization activities, community air monitoring plan, quality assurance project plan, and implementation schedule. This HASP is incorporated into the Work Plan as Appendix A.

Fieldwork will be undertaken to meet requirements outlined in the Work Plan. Field activities may include but are not limited to the following:

- Perimeter and personal air monitoring activities
- Soil borings will be advanced across OU4 for characterization purposes. The Work Plan details specifics regarding field and chemical analysis methods.
- Soil will be generated as a result of drilling and surface soil sampling and will need to be managed appropriately.
- Soil, sediment, and surface water waste disposal sampling will occur as described in the Work Plan.
- Compliance activities, OU4 walks, observations, and other miscellaneous activities.

During field activities, drilling equipment and heavy machinery may be used for characterization purposes. Drilling equipment on OU4 at any time may include Geoprobe™ or similar direct push rig and/or hollow stem auger.

Safety considerations when working around drill rigs and heavy machinery are presented in subsequent sections.

1.2 ORGANIZATION

This HASP covers a broad range of field activities as outlined in preceding sections. Chemical and physical hazard evaluations are presented in Sections 2 and 3, respectively. Specific health and safety guidelines associated with each task, including a brief description of the work, are discussed in Section 11 (Task-Specific Safety Procedures).

1.3 ROLES AND RESPONSIBILITIES

All Integral personnel, subcontractors, or consultants and visitors on OU4 must comply with the requirements of this HASP. The specific responsibilities and authority of management, safety and health, and other personnel are detailed in the following paragraphs.

1.3.1 OU4 Safety Officer

The SSO has full responsibility and authority to implement this HASP and to verify compliance. The SSO reports to the project manager and is on OU4 or readily accessible to the OU4 during all work operations. The SSO is responsible for assessing OU4 conditions and directing and controlling emergency response activities. The specific responsibilities of the SSO include:

- Managing the safety and health functions on OU4
- Serving as the point of contact for safety and health concerns
- Assessing OU4 conditions for unsafe acts and conditions and ensuring corrective action
- Ensuring that all Integral employees and subcontractors understand and follow the HASP
- Ensuring that daily work schedules and tasks are reasonable for the required levels of effort and weather conditions
- Confirming local emergency response phone numbers and locations
- Conducting and documenting the initial and daily or periodic health and safety briefings
- Evaluating and modifying the level of protective apparel and safety equipment, based on OU4 conditions
- Ensuring that the field team observes all necessary decontamination procedures.

If the SSO determines that OU4 conditions are unsafe, he or she has the authority to suspend field operations until the problem is corrected. The SSO can modify HASP procedures in the field. Any changes must be documented in the field logbook, and field staff must be immediately informed of the change. The project manager and Integral's CHSM must be notified by phone or email within 24 hours of any major changes to the HASP.

1.3.2 Project Manager

The project manager has overall responsibility to ensure that personnel working at OU4 are safe. The specific responsibilities of the project manager include:

- Ensuring that the HASP is developed prior to the fieldwork or OU4 visit
- Reviewing and approving the HASP prior to the fieldwork or OU4 visit
- Ensuring employee understanding of and compliance with the HASP.

1.3.3 Corporate Health and Safety Manager

The CHSM provides guidance to the project manager and SSO on HASP preparation and reviews and approves the HASP. The CHSM also serves as an arbitrator if there is a conflict between the project manager, SSO, and field personnel. In addition, the CHSM[1] conducts periodic unannounced audits of Integral field operations to ensure compliance with the HASP.

1.3.4 Field Personnel

All Integral personnel and subcontractors on OU4 are responsible for reading and complying with this HASP, using the proper personal protective equipment (PPE), reporting unsafe acts and conditions, and following the work and safety and health instructions of the project manager and SSO. All Integral personnel, subcontractors, or consultants can and are encouraged to suspend field operations if they feel conditions have become unsafe.

1.4 OU4 DESCRIPTION

OU4 includes areas along the eastern and southern boundaries of the Study Area, consisting of the earthen berm and lands between the eastern boundary of OU1, OU2, OU3, and Post Creek and Highway 17/Interstate 86 to the east, as well as the berm and lands between the southern boundary of OU3 and the Chemung River.

A detailed OU4 map is provided in Attachment 1 to this HASP.

1.5 PROJECT MANAGER AND OTHER KEY CONTACTS

	Name (Affiliation)	Work Telephone	Cell Phone
Project Manager	Jeff Marsh (Integral)		(315) 651-2020
OU4 Safety Officer	TBD (Integral)		
Corporate Health and Safety Manager	Matt Behum (Integral)	(410) 573-1982	(443) 454-1615
Facility Contact	TBD		
Client Contact	TBD		

[1] The audit task may be delegated to an office health and safety representative by the CHSM.

2 CHEMICAL HAZARD EVALUATION

The Final Decision Documents for Study Area OU1, OU2, OU3, and OU5 identified the contaminants of concern (COCs) for the Study Area characterization activities as lead, cadmium, arsenic, and semivolatile organic compounds.[2]

The following table lists the properties of COCs, sample preservatives and decontamination chemicals that may be used at OU4 (i.e., hydrochloric acid, methyl alcohol/methanol, Alconox®, etc.). The table also lists the chemical properties and OSHA permissible exposure limit (PEL), short-term exposure limit (STEL), and immediately dangerous to life and health (IDLH) level. Some chemicals used during equipment decontamination or sample preservation may volatilize and enter the field crew's breathing zone and be inhaled. Breathing zone air can be monitored to ensure that the chemicals do not exceed the PEL. If any of the chemicals exceed the PEL, immediate action is required (e.g., don respirators, leave OU4) as designated in the "Air Monitoring" section (Section 5) of this HASP.

[2] NYSDEC. 2017. Decision Document. Study Area Operable Units (OU) 1, 2 and 5. New York State Department of Environmental Conservation. July 2017. 18 pp.
NYSDEC. 2017. Decision Document. Study Area Operable Unit (OU) 3. New York State Department of Environmental Conservation. July 2017. 18 pp.

Chemical Properties

Chemical of Concern	Maximum Expected Concentration	Medium	PEL/REL (mg/m^3)	OSHA STEL (mg/m^3)	OSHA IDLH (mg/m^3)	IP (eV)	Carcinogen or Other Hazard
Arsenic (inorganic)	Unknown	Soil/Sediment	0.010 (NIOSH TWA REL, 0.002 ceiling)	--	5	--	Ca
Cadmium (inorganic)	Unknown	Soil/Sediment	0.005 (OSHA TWA PEL)	--	9	--	Ca
Hydrochloric Acid	Product (<10%)	Preservative	5	--	50	12.74	Corrosive
Lead (inorganic)	Unknown	Soil/Sediment	0.05 (OSHA TWA PEL)	--	100	--	Irritant, possible carcinogen
Methanol	Product (<62%)	Preservative	200 (OSHA TWA PEL)	250 (NIOSH STEL)	6,000	10.84	Class IB flammable liquid
Nitric Acid	Product (<10%)	Preservative	2	4 (NIOSH STEL)	25	11.95	Corrosive
Alconox® (tetra sodium pyrophosphate)	Product	Decon	5 (NIOSH REL)	--	--	--	Irritant
Isobutylene gas	Product	Calibration Gas	--	--	--	9.43	Irritant

Notes:

-- = none established
Ca = carcinogen
Decon = decontamination
IDLH = immediately dangerous to life and health
IP(eV) = ionization potential (electron volts)
mg/kg = milligrams per kilogram
NA = not available
NIOSH = National Institute for Occupational Safety and Health
PEL = permissible exposure limit
mg/m^3 = milligrams per cubic meter
REL = recommended exposure limit
STEL = short-term exposure limit
TWA = time-weighted average

The table below summarizes the chemical characteristics and potential chemical exposure routes at OU4.

	Likely	Possible	Unlikely
Potential Chemical Exposure Routes at OU4:			
Inhalation		X[a,b,c]	
Ingestion			X[a,b,c]
Skin absorption		X[a,b,c]	
Skin contact		X[a,b,c]	
Eye contact		X[a,b,c]	
Chemical Characteristics:			
Corrosive	X[a]		X[b,c]
Flammable	X[b]		X[a,c]
Ignitable	X[b]		X[a,c]
Reactive	X[a]		X[b,c]
Volatile	X[a,b]		X[c]
Radioactive			X[a,b,c]
Explosive			X[a,b,c]
Biological agent			X[a,b,c]
Particulates or fibers		X[c]	X[a,b]
If likely, describe:	Sample preservatives may include hydrochloric acid, methyl alcohol/methanol, and nitric acid. These are used for sample preservation in small volumes. Methyl alcohol/methanol is volatile and flammable. Field personnel will stand upwind when using methyl alcohol. These chemicals will not be used unless area is well ventilated. Keep methyl alcohol away from ignition sources at all times. Avoid contact with skin and eyes. Nitric and hydrochloric acids are corrosive and volatile. Always wear goggles or safety glasses and nitrile gloves when filling preserved bottles. These chemicals will not be used unless area is well ventilated.		

Notes:

[a] Nitric and hydrochloric acid (preservatives).

[b] Methyl alcohol (preservative).

[c] OU4 chemicals.

3 PHYSICAL HAZARD EVALUATION AND GUIDELINES

The following sections present general physical hazards and soil and sediment sampling guidelines.

3.1 GENERAL PHYSICAL HAZARDS

The following table presents possible physical hazards that are expected to be present during field activities.

Possible Hazard	Yes	No	Proposed Safety Procedure
Heavy equipment	X		Stay back from operating equipment; wear safety vests and hard hats; coordinate and maintain eye contact with equipment operator. Large haul trucks may be on OU4 and have limited visibility. Be sure to maintain a safe distance from haul truck routes and maintain eye contact with the driver, or wave your hands to get their attention when walking around a truck being loaded. Be sure to make sure the operator acknowledges that you are seen.
Material handling	X		Lift properly; seek assistance if necessary; do not overfill coolers or boxes. Seek assistance if drums must be moved.
Adverse weather	X		Seek shelter during electrical storms; work in adverse weather conditions only with proper training and equipment.
Plant/animal hazards	X		Know local hazards and take appropriate precautions. Use insect repellent if mosquitoes are persistent.
Dust	X		Wet surfaces/work areas, reduce truck speeds, and stand upwind.
Hazardous material handling (sample preservative)	X		Wear proper PPE and do not allow volatile components to enter your breathing zone; work in well ventilated areas and upwind.
Uneven terrain/tripping	X		Use caution, wear properly fitting shoes or boots, and keep work area orderly. Do not obscure your view of the ground with carried loads
Noise	X		Wear ear protection when working around heavy equipment and other noise sources. Excavators, rock breaking equipment, and haul trucks are loud enough to damage your hearing. Wear hearing protection at all times when around heavy machinery.
Heat stress	X		Follow heat stress information (Attachment 3). *Note:* potential for heat stress will depend on ambient temperatures, PPE in use, activities, hydration, etc.
Cold/hypothermia	X		Keep warm and dry; bring changes of clothes; do not work in extreme conditions without proper equipment and training. Follow cold stress information (Attachment 3).

Possible Hazard	Yes	No	Proposed Safety Procedure
Falling objects	X		Wear hard hats in work areas (i.e., to protect from overhead hazards, mainly associated with operation of heavy equipment).
Drill rigs	X		Avoid all pinch points; do not operate or stand near rig during electrical storms; stay a safe distance (25 ft) from power lines; level drill rig.
Work near water	X		Pursuant to OSHA 1926.106(a)&(b), all staff working onsite in or near water will be required to wear buoyant work vests or life preservers, either of which will be inspected before and after use for defects that would alter strength and buoyancy. Defective units shall be immediately discarded and replaced with non-defective units prior to sampling.

Summary of potential physical hazards posed by proposed OU4 activities:

Activity	Potential Hazard
Sample handling	Hazardous material handling (sample preservatives), uneven terrain and tripping, heat stress, hypothermia, adverse weather, plant and animal hazards, drill rigs.
Air monitoring and observations	Heavy equipment, falling objects, uneven terrain and tripping, heat stress, hypothermia, adverse weather, excavations, plant and animal hazards, noise, dust.

4 PERSONAL PROTECTIVE EQUIPMENT AND SAFETY EQUIPMENT

The following sections address PPE and safety equipment required for completing the field activities.

4.1 PERSONAL PROTECTIVE EQUIPMENT

Based on the hazards identified above in Sections 2 and 3, the following table identifies the PPE required for OU4 activities.

	Level of Protection	
OU4 Activity	Initial	Contingency[a]
Air and soil sampling	D/MD	Temporarily stop work and assess situation[a]
Sample handling and decontamination	D	Temporarily stop work and assess situation[a]
Drilling oversight and core logging	D/MD	Temporarily stop work and assess situation[a]

Notes:
[a] Based on unexpected change in OU4 conditions

Each level of protection will incorporate the following PPE:

Level D	Long pants or work coveralls, shirt, hard hat (if heavy machinery is present at OU4 or when working near overhead hazards), traffic safety vest (if heavy machinery is present), latex or nitrile gloves, safety glasses, and steel-toe boots are required. Hearing protection, sunscreen, and rain gear are required as needed.
Level MD	Same as Level D with addition of rain gear and/or chest waders and personal flotation device, as necessary.

Respirator and Respirator Cartridge Information

Respirator use is not expected to be necessary for this project. However, there is potential to encounter volatile organic compounds (VOCs) and other unknown hazards as characterization of OU4 proceeds. If unexpected conditions are encountered resulting in an exceedance of the action level (see Section 5.4 below), work will be stopped, the situation assessed, and engineering controls potentially implemented.

If it is determined that respirators will need to be worn on specific portions of OU4, change-out schedules and procedures for respirator use will be incorporated as an addendum to this HASP. Change-out schedules and calculation parameters for many chemicals can be calculated using the cartridge life calculator at the Mine Safety Appliances Company website (http://www.msanorthamerica.com/).

4.2 SAFETY EQUIPMENT

The following safety equipment will be present at OU4 during the proposed field activities.

Air Monitoring (check the items required for this project)

- [ ] OVM
- [ ] LEL/O_2 meter
- [ ] H_2S meter
- [ ] Detector pump and tubes (e.g., benzene)
- [X] PID
- [X] Miniram (particle monitors)
- [ ] Radiation meter
- [X] Other: Personal Flotation Device

First Aid Kit Mandatory, including absorbent compress, adhesive bandages, adhesive tape, antiseptic, burn treatment, medical exam gloves, sterile pad, cardiopulmonary resuscitation (CPR) shield, triangle bandage, scissors—for cutting off the PPE from an injured person (check additional items required)

- [X] Emergency blanket
- [X] Insect repellent
- [X] Sunscreen
- [ ] Other: _____

Other (check the items required for this project)

- [X] Eyewash
- [X] Drinking water
- [ ] Stopwatch for monitoring heart rate for heat stress monitoring[3]
- [ ] Thermoscan® thermometer for heat stress monitoring
- [ ] Survival kit[4]
- [X] Personal flotation device
- [ ] Fit test supplies
- [X] Fire extinguisher (drill rigs and onboard larger sampling vessels)
- [ ] Windsock
- [X] Cellular phone
- [ ] Radio sets
- [X] Global positioning system
- [ ] Other: _____

[3] Heart rate monitoring requires special training.
[4] Consult the CHSM for guidance for Subject Property-specific survival kits.

5 AIR MONITORING

This section covers personal air monitoring for field personnel. A community air monitoring plan is included as Appendix B to the Work Plan. Air monitoring will be conducted when entering previously uncharacterized areas, when working in the vicinity of uncontained chemicals or spills, when opening containers and well casings, and prior to opening confined spaces. (Note: Integral personnel are not trained or authorized to enter confined spaces under any circumstances.) Air monitoring must be conducted to identify potentially hazardous environments and determine reference or background concentrations. Air monitoring can sometimes be used to augment judgment in defining exclusion zones.

Air monitoring may be discontinued at locations where there have been multiple sampling events in the same area/media during similar activities with no action level exceedances. In such instances, the air monitoring results must be well documented and there must be approval from the CHSM prior to discontinuing the air monitoring. Air monitoring must be reinstated for fieldwork in different areas of OU4 or when sampling new media.

5.1 INTRODUCTION

Personal air monitoring involves collection of samples within the breathing zone of the field personnel to better understand exposures, ensure appropriate levels of PPE, and document compliance with regulation. Such samples may be full shift, for comparison to PELs (or other applicable occupational exposure limits), or short term, for comparison to STELs. Some chemicals in soil or aqueous media may volatilize or become aerosolized and be inhaled by field personnel.

Breathing zone air can be monitored to ensure that the chemicals do not exceed a regulatory or project-specific action level (generally 50 percent of the PEL). Integral commonly uses photoionization detectors (PIDs) and dust meters (e.g., MiniRam) for monitoring VOCs and particle constituents, respectively. In practice, the air directly in the field personnel's breathing zone is monitored with the PID or dust meter for 10–15 seconds. The highest reading is recorded in the project logbook and checked against the OU4-specific action level in the table below. If any of the constituents exceed the action level presented in Section 5.4, immediate action is required (e.g., don respirators, leave OU4, etc.), as designated.

The following sections provide general guidance on the selection and calibration of PIDs and dust meters, which are typically rented for field projects.

5.2 PHOTOIONIZATION DETECTORS

It is critical to order a PID with a detector lamp with the appropriate ionization energy to detect constituents of interest at OU4. The ionization energy of the lamp must be greater than the ionization potential of the constituents of interest (ionization potentials are listed in the National Institute for Occupational Safety and Health pocket guide to chemicals and are presented in Section 2). Be sure that the meter arrives at least a day prior to the start of the fieldwork so field personnel can familiarize themselves with the operation of the meter and confirm that it was not damaged during shipping. Field personnel must also read the operation manual to become familiar with its operation prior to use in the field. Note that moisture may damage the detector lamp and/or provide erroneous readings, so a moisture filter is used on the probe. Also note that the PID will only accurately quantitate the material used in the calibration process. A response factor is used to measure the sensitivity of the PID to a particular chemical present at OU4. Response factors are normally presented in the operation manual for the PID.

As VOCs with a higher ionization energy are not initially expected to be of concern at OU4, a 10.6 eV lamp PID will be used unless site observations and/or data indicate that a lamp with a higher ionization energy needs to be used.

The PID must be calibrated daily in accordance with the manufacturer's specifications, which are provided in the operation manual. The calibration typically requires the use of a span gas (generally 100 parts per million isobutylene) and zero gas (generally fresh air). Be sure that all the required calibration equipment/supplies are provided with the PID (e.g., span gas cylinder, regulator, tubing, and Tedlar™ bag). Record calibration data in the field logbook.

5.3 DUST METERS

The principal particle size of concern at OU4 is PM10. Air monitoring will be performed with a MiniRam or equivalent device, which is capable of detecting PM10 in air.

It is critical that the dust meter is capable of measuring the concentrations of airborne dust that are at or below OU4-specific action levels presented below. Be sure that the meter arrives at least a day prior to the start of the fieldwork so field personnel can familiarize themselves with the operation of the meter and confirm that it was not damaged during shipping. Field personnel must also read the operation manual to become familiar with its operation prior to use in the field.

The dust meter must be field checked (i.e., zeroed) daily in accordance with the manufacture's specifications, which are provided in the operation manual. The dust meter field check typically involves zeroing the meter with ambient or filtered air. Be sure that all the required zeroing and operational equipment/supplies are provided with the dust meter. Record field-check data in the field logbook.

5.4 ACTION LEVELS

The following is a summary of personal air monitoring to be conducted at OU4.

Instrument	Observation	Action	Comments
PID	<5 ppm	Continue working.	At the boring/sampling location or OU4 perimeter and sustained for 5 minutes.
PID	≥5 ppm	Work will stop and operations will be reviewed.	Steps will be taken to reduce emissions, such as placement of tarps or suppressants over the open work area, and the areas will be retested.
MINIRAM	≤50 µg/m^3	Continue working.	
MINIRAM	>50 µg/m^3 (At the boring/sampling ocation or OU4 perimeter and sustained for 5 minutes)	Implement additional dust control measures.	
MINIRAM	>150 µg/m^3 at OU4 property perimeter and sustained for 5 minutes)	Operations will temporarily cease while additional dust control measures are identified and implemented.	If >150 µg/m^3 is detected at the perimeter of OU4, operations will slow while the optimal additional dust control measures are identified.

Note:

ppm = parts per million
µg/m^3 = micrograms per cubic meter

Air monitoring will be conducted at least every 30 minutes, or more frequently if odors are observed by the field crew. Maintenance and calibration and field checks of all air monitoring equipment will be performed in accordance with manufacturer recommendations. Further details regarding community air monitoring are provided in the community air monitoring plan (Appendix B to the Work Plan).

6 HEALTH AND SAFETY TRAINING AND MEDICAL MONITORING

The following sections present requirements for health and safety training and medical monitoring.

6.1 Health and Safety Training and Medical Monitoring

Integral and subcontractor personnel are required to complete the following training requirements prior to working at OU4.

6.1.1 Training Requirements

Task	No Training	24-hour	40-hour[a]	Supervisor[b]	First Aid/CPR[c]	Medical Monitoring
Integral Field Personnel						
TBD			X	X	X	X
TBD			X	X	X	X
TBD			X	X	X	X
Integral Subcontractors[d]						

Notes:

[a] Must have current OSHA 8-hour refresher if it has been more than a year since the OSHA 40-hour training.

[b] At least one person at OU4 must be OSHA HAZWOPER supervisor trained if this is a hazardous waste site.

[c] At least one member of each team of two or more people at OU4 must be First Aid/CPR trained.

[d] Integral subcontractors and consultants may have requirements that are more stringent than those listed above. These are minimum training and monitoring requirements required to work at OU4.

6.1.2 OU4 Safety Meetings

OU4 safety meetings must be held before beginning new tasks or when new staff enter OU4. OU4 safety meetings should be held at a minimum of once a week and should be held daily on complex or high hazard projects. Tailgate safety meetings must occur every morning during review of the day's work plan, covering specific hazards that may be encountered. Additional meetings will be held at any time health and safety concerns are raised by any of the personnel. Attendance and topics covered are to be documented in the field logbook.

6.2 MEDICAL MONITORING

OSHA requires medical monitoring for personnel potentially exposed to chemical hazards in concentrations in excess of the PEL for more than 30 days per year and for personnel who must use respiratory protection for more than 30 days per year. Integral requires medical monitoring for all employees potentially exposed to chemical hazards.

Will personnel working at OU4 be enrolled in a medical monitoring program?	Yes	X
	No	

7 EMERGENCY RESPONSE PLAN

The following sections discuss emergency recognition and prevention, emergency response and notification, emergency decontamination, OU4 communications, and use of the buddy system.

7.1 EMERGENCY RECOGNITION AND PREVENTION

It is the responsibility of all personnel to monitor work at OU4 for potential safety hazards. All personnel are required to immediately report any unsafe conditions to the SSO. The SSO is responsible to immediately take steps to remedy any unsafe conditions observed at OU4.

The following are examples of some emergency situations that could occur during the characterization field activities:

- Slips, trips, and falls (on sloped areas, uneven terrain, near Post Creek and Chemung River, etc.)
- Lacerations from scrap metal (in soil, etc.)
- The air monitoring action level is exceeded
- Entrainment of clothes or objects in moving equipment or parts
- Serious injury or illness (e.g., physical injury, heart attack)
- Severe thunderstorm with lightning.

Immediate actions will be taken by the field team under the leadership of the SSO in response to these emergencies.

7.2 EMERGENCY RESPONSE AND NOTIFICATION

If an emergency at OU4 warrants it, all personnel must immediately evacuate the affected work area and report to the SSO at the predetermined emergency assembly location:

Field vehicle

In case of injury, field personnel should take precautions to protect the victim from further harm and notify local or facility emergency services. In remote areas, it will be necessary to have first aid-trained personnel on the field team. The victim may require decontamination prior to treatment if practicable—requirements will vary based on OU4 conditions.

Emergency medical care will be provided by:

- [X] Local emergency medical provider (i.e., fire department)
- [ ] Facility emergency medical provider
- [ ] First aid-trained field staff (for remote areas only)

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
Fire	Corning Fire Department	911	No
Police	Corning Police Department	911	No
Ambulance	Not available	911	No
Hospital	Guthrie Corning Hospital	607-937-7200	No
Directions to the hospital:	Consult attached hospital route map.		

The SSO must confirm that the hospital listed is still in operation and that it has an emergency room. **It is required that the SSO drive to the hospital so that the directions are practiced and understood prior to initiating fieldwork.**

Corporate Resource	Name	Work Telephone	Cell Phone
Integral CHSM[a]	Matt Behum	Office: (410) 573-1982	(443) 454-1615
Integral President	Bill Locke	Office: (720) 465-3315	(303) 548-1111
Integral Human Resources Manager	Joseph Drew	Office: (206) 957-0330	(206) 379-1289
Medical consultant	Dr. Peter Greaney (WorkCare)	Office: (800) 455-2219 x 2114	NA

Notes:

[a] If the CHSM cannot be reached, call Eron Dodak [Office: (503) 943-3614; Cell: (503) 407-2933]. If Eron Dodak cannot be reached, call David Livermore [Office: (503) 943-3613; Cell: (503) 806-4665].

In case of serious injuries, death, or other emergency, the Integral CHSM must be notified immediately at the phone numbers listed above. The Integral CHSM will notify the project manager and Integral's President. The project manager will notify the client.

7.3 EMERGENCY DECONTAMINATION PROCEDURES

In case of an emergency, if possible, gross decontamination procedures will be promptly implemented. If a life-threatening injury occurs and the injured person cannot undergo decontamination procedures at OU4, then the medical facility will be informed that the injured

person has not been decontaminated and given information regarding the most probable chemicals of concern.

Decontamination procedures will only be used if practical and if they will not further injure the person or delay treatment. Decontamination procedures should not be implemented if there is not a reasonable possibility that the injured party requires such intervention. The SSO will make the determination whether or not to decontaminate the injured person. The following steps will be followed for decontaminating injured personnel while in OU4:

- If it will not injure the person further, cut off PPE using scissors or scrub the gross contamination from the injured person's PPE (e.g., Tyvek® coveralls, work boots) with a Liquinox® or Alconox® solution followed by a rinse with tap or deionized/distilled water.
- Remove PPE if feasible without further injuring the person.

7.4 OU4 COMMUNICATIONS

Each field team will carry a cell phone or satellite phone that is in good working order. If there is any type of emergency that requires OU4 to be evacuated (e.g., severe thunderstorm with lightening, chemical release), the field team leader will blow an air horn three times. When the horn sounds, all personnel will meet at the predetermined emergency assembly location, provided the muster point is in safe territory (field vehicle). All other emergency notifications that do not require evacuation (e.g., a person falling overboard) will be conducted using a cell or satellite phone. Emergency phone numbers are listed above in Section 7.2.

7.5 BUDDY SYSTEM

The buddy system will be used at OU4 at all times. The buddy system is a system of organizing employees into field teams in such a manner that each employee of the field team is designated to be observed by at least one other employee in the field team. The purpose of the buddy system is to provide rapid assistance to employees in the event of an emergency.

Integral field staff will always have someone else on OU4 with them. It is Integral's policy that the buddy system will be used at all times. If Integral personnel are unable to team with other Integral staff, contactors will be retained to maintain the buddy system. Working alone at a specific area of OU4 should be avoided given the potential hazards. Sometimes it is necessary for one member of the field team to be temporarily out of the visual sight of the other, and when this must occur, contact will be made with OU4 personnel during regularly agreed upon intervals by cell phone or radio.

8 WORK ZONES

Work zones are defined as follows:

Exclusion zone	Any area of OU4 where hazardous substances are present, or are reasonably suspected to be present, and pose an exposure hazard to personnel
Contamination reduction zone	Area between the exclusion and support zones that provides a transition between contaminated and clean zones
Support zone	Any area of OU4, so designated, that is outside the exclusion and contamination reduction zones

OU4 control measures in work zones are described below for upland sampling and broken further down into specific field activities.

8.1 DRILLING AND SAMPLING

These activities include oversight of the installation of boreholes and associated soil sampling.

Exclusion zone: An approximate 12-ft radius around the drill rig will be marked with orange traffic safety cones or caution tape. Only properly equipped and trained personnel (i.e., wearing modified D protective clothing) will be allowed in this area.

Contamination reduction zone: After drilling and/or sampling is completed at a station, the exclusion zone will become the contamination reduction zone.

Support zone: All areas outside the exclusion and contamination reduction zones.

Controls to be used to prevent entry by unauthorized persons: No unauthorized personnel will be allowed into the exclusion/contaminant reduction zones.

8.2 Sediment and Surface Water Sampling

Exclusion zone: An approximate 12-ft radius, or any in-stream area where water is present, will be considered the exclusion zone. Sample collection and processing will occur in this area. Only properly equipped and trained personnel (i.e., wearing modified D protective clothing) will be allowed in this area.

Contamination reduction zone: After sediment sampling is completed at a station, the exclusion zone will become the contamination reduction zone.

Support zone: All areas outside the exclusion and contamination reduction zones.

9 EQUIPMENT DECONTAMINATION AND PERSONAL HYGIENE

The following sections describe equipment decontamination and personal hygiene procedures.

9.1 EQUIPMENT DECONTAMINATION PROCEDURES

After sampling is completed, the exclusion zone will be used as the contaminant reduction zone for decontamination activities.

To minimize or prevent personal exposure to hazardous materials, all personnel working in the exclusion zone and contamination reduction zone will comply with the following decontamination procedures:

- All personnel will wash soil and chemicals from their raingear or clothing before leaving the exclusion zone. All gloves, rain gear, and outer boots will be removed prior to entering the field vehicle. When that is impractical, lay down plastic sheeting over the seat and use a disposable floor mat or plastic sheeting for the floor of the vehicle to reduce the possibility of soiled material becoming adhered to the interior of the car.

Decontamination equipment required at OU4 includes the following:

- Buckets or tubs
- Distilled/deionized water
- Scrub brushes (long-handled)
- Liquinox® or Alconox® detergent
- Plastic bags
- Foil
- Paper towels
- Garbage bags
- Clean garden sprayer.

All non-disposable components of the sampling equipment (e.g., stainless steel spoons and bowls used for sample compositing) that contact the soil or sediment will be decontaminated using the following steps:

1. Rinse with tap water
2. Wash with Alconox® or Liquinox® detergent
3. Rinse with tap water

4. Rinse with distilled/deionized water using a garden sprayer (compositing equipment only)
5. Allow to air dry
6. Wrap up compositing equipment in aluminum foil or place in a sealed plastic bag.

Decontamination wastewater containing solvent rinsate will be collected in plastic tubs and allowed to evaporate in an area downwind of the field crew during the course of the decontamination activity. Any solvent rinsate that has not evaporated by the end of the decontamination activity will be containerized and disposed of in accordance with applicable regulations.[5]

9.2 PERSONAL HYGIENE

The following personal hygiene practices will be used at OU4 to reduce exposure to chemicals.

- Long hair will be secured away from the face so it does not interfere with any activities.
- All personnel leaving potentially contaminated areas will wash their hands, forearms, and faces in the contaminant reduction zone prior to entering any clean areas or eating areas.
- Personnel leaving potentially contaminated areas will shower (including washing hair) and change to clean clothing as soon as possible after leaving OU4.
- No person will eat, drink, or chew gum or tobacco in potentially contaminated areas. Single portion drink containers and drinking of replacement fluids for heat stress control will be permitted only in support areas.
- Smoking is prohibited by Integral personnel and subcontractors in all areas of OU4 because of the potential for contaminating samples and for the health of the field team.

[5] Integral personnel are not allowed to sign hazardous waste manifests. Hazardous waste manifests must be signed by the client or client's representative.

10 VEHICLE SAFETY, SPILL CONTAINMENT, AND SHIPPING INSTRUCTIONS

The following sections describe vehicle safety, spill containment, and shipping instructions.

10.1 VEHICLE SAFETY

Integral's vehicle safety program requires the following:

- Cell phone usage while driving is not allowed, including the use of hands-free devices. If it not feasible to wait to use the cell phone until arriving at the destination, pull off the road and park in a safe location to use the cell phone. Do not pull to the side of the road to use a cell phone because this significantly increases the risk of a rear-end collision.
- All vehicles are to be operated in a safe manner and in compliance with local traffic regulations and ordinances.
- Drivers are to practice defensive driving and drive in a courteous manner.
- Drivers are required to have a valid driver's license and liability insurance (per local state laws).
- Seat belts are to be worn by the driver and all passengers.
- No persons are allowed to ride in the back of any trucks or vans, unless equipped with seatbelts.
- Vehicles are to be driven in conformance with local speed limits.
- Personnel who are impaired by fatigue, illness, alcohol, illegal or prescription drugs, or who are otherwise physically unfit, are not allowed to drive or work on Integral field locations.
- Personnel are to avoid engaging in other distractions such as changing radio stations while driving.
- Motor vehicle accidents are to be reported to the responsible law enforcement agency, the Integral human resources manager, and the Integral CHSM on the same day of occurrence. Documentation of damage should be photographed.
- Personnel who have experienced work-related vehicle accidents or citations may be required to complete a defensive driving program.

10.2 SPILL CONTAINMENT

Decontamination chemicals to be used at OU4 will be either Liquinox® or Alconox®. These chemicals will be dispensed from capped or disposable containers directly into plastic pails or shallow Rubbermaid® tubs that are marked as used for decontamination. Plastic sheeting should be laid down beneath the decontamination buckets and care will be taken to reduce spillage and splashing during decontamination. Any spills will be cleaned up and disposed of in accordance with applicable regulations.

10.3 SHIPPING INFORMATION

Federal laws and international guidelines place restrictions on what materials may be shipped by passenger and cargo aircraft. In addition, 49 CFR regulates labeling, manifesting, and shipment of all packages containing potentially hazardous materials. In the course of these field activities, the following items will be shipped to and from OU4 as shown below:

Item	Hazardous Constituent	Quantity	Packaging	How Shipped
Soil samples	None	Approx. 100	Coolers	Field vehicle or courier
Calibration gas (isobutylene)	Isobutylene	(1) 17 L	Steel cylinder	Field vehicle

A 24-hour emergency response number (on any shipping documents such as a Uniform Hazardous Waste Manifest, Shipper's Declaration of Dangerous Goods, etc.) is required for shipments of all dangerous or hazardous goods. Integral does not have a 24-hour emergency contact number for dangerous or hazardous goods shipment. No dangerous or hazardous goods may be shipped by Integral until an account is set up with a 24-hour emergency response service such as CHEM-TEL (1-813-248-0573). If any hazardous or dangerous goods need to be shipped for a project, they must be shipped directly to OU4 by the supplier. Any hazardous or dangerous goods that are not used in the course of the field effort must remain at OU4.

The samples will be prepared and labeled for shipment in accordance with the sampling and analysis plan developed for OU4.

Air shipment of equipment with lithium batteries is required to note the presence of these batteries. Warning labels are available from the equipment rental agency and can be copied.

Do not ship any isobutylene containers (empty or not) back to the vendor.

11 TASK-SPECIFIC SAFETY PROCEDURE SUMMARY

The following sections briefly describe general procedures, and task-specific upland, sediment, and surface water sampling safety procedures.

11.1 GENERAL PROCEDURES

The following safety procedures are applicable to all OU4 activities.

11.1.1 Trespassers

Trespassing may be a concern at OU4. Always use the buddy system at OU4. Personnel should avoid trespassers, if possible, and not actively engage with trespassers unless the trespasser is affecting the work activity, and/or to avoid hostility. If a situation occurs that results in an unsafe confrontation, personnel should immediately enter a locked vehicle, leave OU4, and call law enforcement authorities.

11.1.2 Weather Extremes

Fieldwork may occur during warm temperatures. An information sheet on heat stress is included as Attachment 3 and should be reviewed prior to working in conditions where heat stress is a potential risk to personnel.

11.2 UPLAND SOIL SAMPLING

The Dig Safely, New York one-call utility locating service (1- 800-962-7962) will be notified at least 48 hours (2 full working days) prior and not more than 10 days prior to any subsurface characterization work. Confirm the absence of underground and overhead utilities before starting subsurface activities. Assure that all utilities are marked or have a designation that they are not present in the area. The utility locating service should have marked all utilities present in the area. It may be necessary to hire a private utility locator as work occurring on private property may not be adequately marked. Take a few minutes to examine the locations of fire hydrants, gas meters, etc. to make sure that the utility locating marks make sense. If there is any doubt as to the location of underground utilities, call the public or a private utility locator. Finally, check for overhead utilities and obstructions such as trees.

Personnel will wear safety glasses and steel toed boots at all times. Hard hats and traffic safety vests will be worn when heavy equipment or drill rigs are present, or where overhead hazards exist. The exclusion zone around the drill rig or excavation will be marked with orange traffic cones or caution tape, as practicable, and personnel will police the area to make sure no

unauthorized personnel enter the exclusion zone. Avoid getting soil and sample preservatives (hydrochloric acid, methanol, and nitric acid) on your clothes or skin. Exercise care when lifting, assembling, and decontaminating equipment. Always stay clear of the drill rig and other heavy machinery. Be aware of your surroundings and understand that blind spots do exist with heavy machinery, so always be aware of their location. Keep in eye contact with the driller and/or equipment operator/driver. Stay away from pinch points. Know the location of the "kill switch" on the rig. Avoid haul truck routes and make sure the operator sees you at all times even when not working in an excavation area. When in an excavation area, ensure that you are well outside of the swing radius of the excavator. While excavation is occurring, ensure airborne dust is kept to a minimum by wetting surfaces to suppress dust.

Exercise caution when working in the floodplain. Always be aware of the surroundings, stream current, and water depths and pay attention to rainfall predictions. Personnel must stop work near the stream if precipitation occurs as water depths can change quickly and unpredictably. If a large storm event is forecasted, work on the floodplain will be postponed until water levels return to safe elevations.

11.3 SEDIMENT AND SURFACE WATER SAMPLING

Always wear a U.S. Coast Guard–approved personal flotation device when doing any work in Chemung River and Post Creek areas with water depths greater than knee high. Personnel may not enter water at a depth greater than waist high at any time. Chest waders will be worn when entering the water for sampling. A hard hat, safety glasses, steel-toe boots, and nitrile gloves are required at all times without exception. Use hearing protection as needed.

Exercise caution when working in stream. Always be aware of the surroundings, stream current, and water depths. Personnel must exit the stream if precipitation occurs as currents and water depths can change quickly and unpredictably. Maintain eye contact with your buddy at all times. Uneven terrain can cause personnel to slip or fall, and rocks, cobbles, and woody debris can entrain or trap personnel. Keep sampling equipment on the shoreline organized at all times.

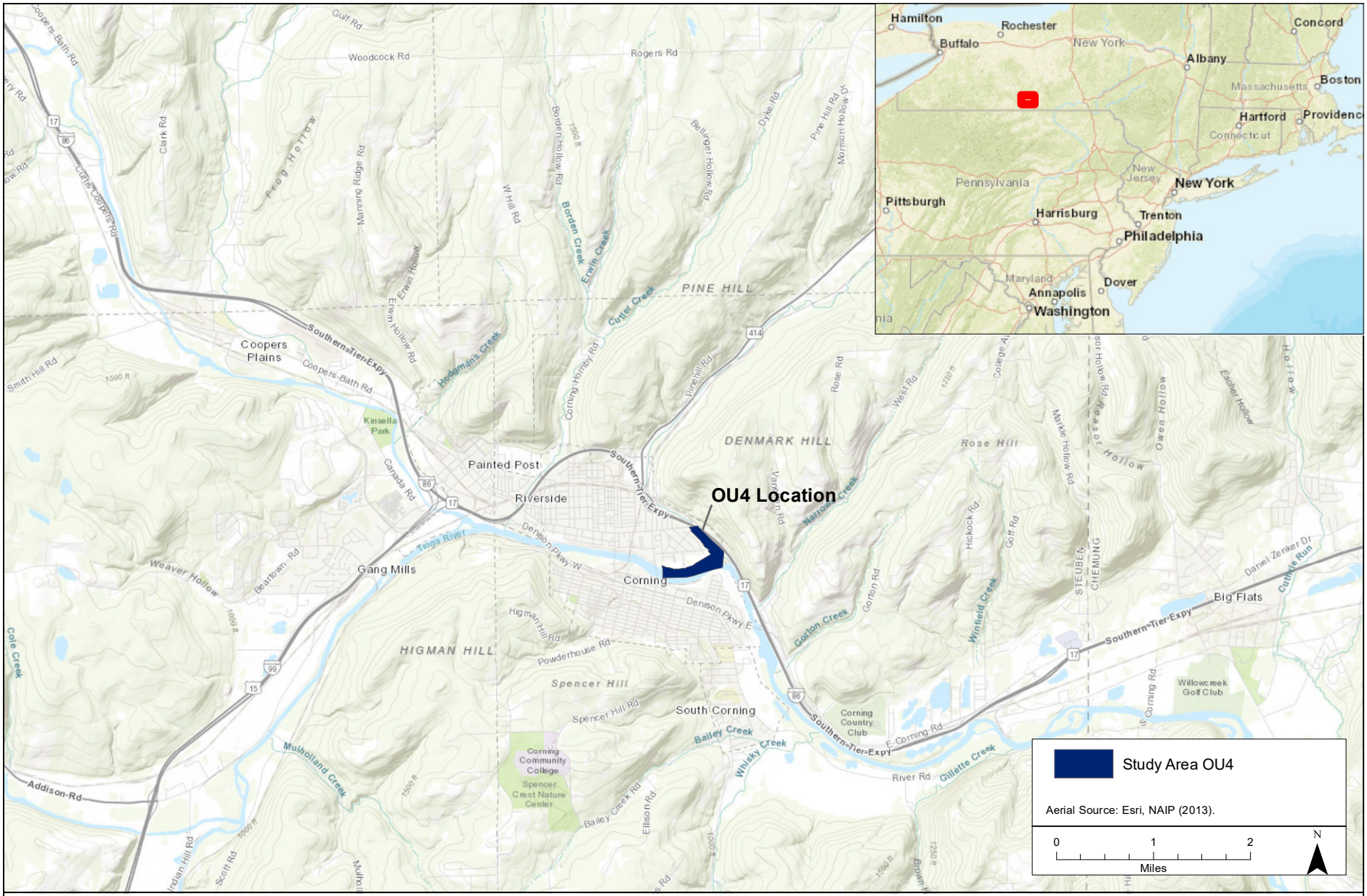
Avoid getting sediment and decontamination chemicals on clothes or skin. Exercise care when lifting equipment and when entering and exiting the stream.

11.4 OBSERVATION AND GENERAL OU4 ACTIVITIES

Be aware of your surroundings and the potential for uneven terrain. Wear appropriate PPE depending on conditions and at all times when sampling. Steel toed boots will be worn at all times while at OU4.

ATTACHMENT 1

STUDY AREA OU4 MAP AND HOSPITAL ROUTE

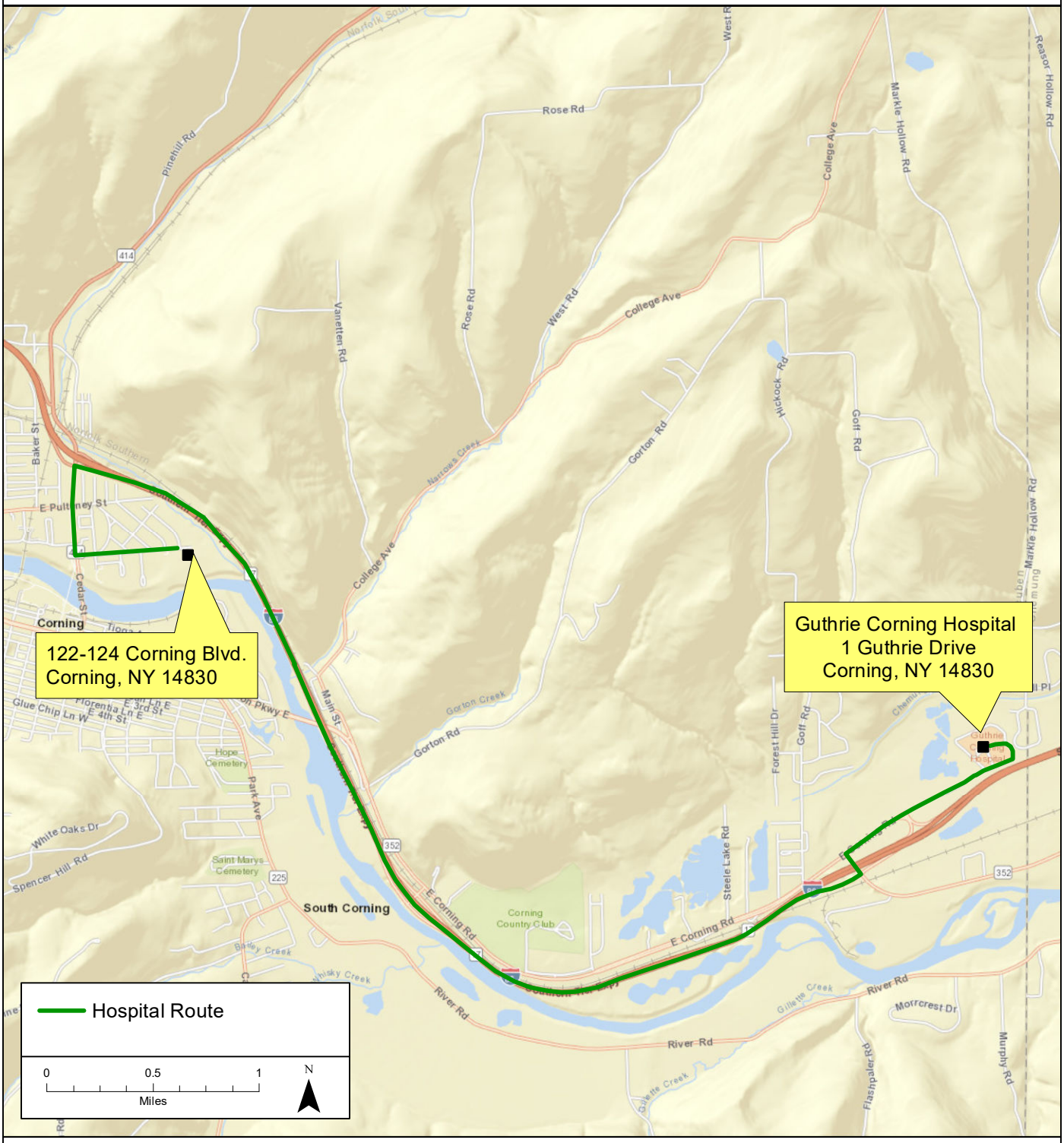


integral engineering pc

Study Area OU4

N:\GIS\Projects\E160_StudyAreaOU4_CI\Working_MXDs\Subject_Property_Map.mxd 4/14/2020 3:55:45 PM

1. Head west on Corning Blvd.
2. Turn right on Center Way.
3. Turn right to merge onto I-86 E/NY-17 E.
4. Take exit 48 for NY-352 toward E Corning.
5. At the top of the ramp, turn left onto NY-352 W.
6. At the stop light turn right.
7. The hospital will be on the left.



N:\GIS\Projects\E160_StudyAreaOU4_CI\Working_MXDs\Hospital Route.mxd 4/14/2020 2:21:40 PM

integral

Hospital Route Map

ATTACHMENT 2

REGULATORY NOTICES

You Have a Right to a Safe and Healthful Workplace.

IT'S THE LAW!

- You have the right to notify your employer or OSHA about workplace hazards. You may ask OSHA to keep your name confidential.
- You have the right to request an OSHA inspection if you believe that there are unsafe and unhealthful conditions in your workplace. You or your representative may participate in the inspection.
- You can file a complaint with OSHA within 30 days of discrimination by your employer for making safety and health complaints or for exercising your rights under the *OSH Act*.
- You have a right to see OSHA citations issued to your employer. Your employer must post the citations at or near the place of the alleged violation.
- Your employer must correct workplace hazards by the date indicated on the citation and must certify that these hazards have been reduced or eliminated.
- You have the right to copies of your medical records or records of your exposure to toxic and harmful substances or conditions.
- Your employer must post this notice in your workplace.

The *Occupational Safety and Health Act of 1970 (OSH Act)*, P.L. 91-596, assures safe and healthful working conditions for working men and women throughout the Nation. The Occupational Safety and Health Administration, in the U.S. Department of Labor, has the primary responsibility for administering the *OSH Act*. The rights listed here may vary depending on the particular circumstances. To file a complaint, report an emergency, or seek OSHA advice, assistance, or products, call 1-800-321-OSHA or your nearest OSHA office: • Atlanta (404) 562-2300 • Boston (617) 565-9860 • Chicago (312) 353-2220 • Dallas (214) 767-4731 • Denver (303) 844-1600 • Kansas City (816) 426-5861 • New York (212) 337-2378 • Philadelphia (215) 861-4900 • San Francisco (415) 975-4310 • Seattle (206) 553-5930. Teletypewriter (TTY) number is 1-877-889-5627. To file a complaint online or obtain more information on OSHA federal and state programs, visit OSHA's website at **www.osha.gov**. If your workplace is in a state operating under an OSHA-approved plan, your employer must post the required state equivalent of this poster.

1-800-321-OSHA
www.osha.gov

U.S. Department of Labor • Occupational Safety and Health Administration • OSHA 3165

ATTACHMENT 3

SAFETY PROCEDURES

FROSTBITE

What happens to the body:

Freezing in deep layers of skin and tissue; pale, waxy-white skin color; skin becomes hard and numb; usually affects fingers, hands, toes, feet, ears, and nose.

What to do: (land temperatures)

- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet or tight clothing that may cut off blood flow to the affected area.
- **Do not** rub the affected area because rubbing damaged the skin and tissue.
- Gently place the affected area in a warm water bath (105°) and monitor the water temperature to **slowly** warm the tissue. Don't pour warm water directly on the affected area because it will warm the tissue too fast, causing tissue damage. Warming takes 25-40 minutes.
- After the affected area has been warmed, it may become puffy and blister. The affected area may have a burning feeling or numbness. When normal feeling, movement, and skin color have returned, the affected area should be dried and wrapped to keep it warm. **Note:** If there is a chance the affected area may get cold again, do not warm the skin. If the skin is warmed and then becomes cold again, it will cause severe tissue damage.
- Seek medical attention as soon as possible.

How to Protect Workers

- Recognize the environmental and workplace conditions that lead to potential cold-induced illnesses and injuries.
- Learn the signs and symptoms of cold-induced illnesses/injuries and what to do to help the worker.
- Train workers about cold-induced illnesses and injuries.
- Select proper clothing for cold, wet, and windy conditions. Layer clothing to adjust to changing environmental temperatures. Wear a hat and gloves, in addition to underwear that will keep water away from the skin (polypropylene.)
- Take frequent short breaks in warm, dry shelters to allow the body to warm up.
- Perform work during the warmest part of the day.
- Avoid exhaustion or fatigue because energy is needed to keep muscles warm.
- Use the buddy system (work in pairs.)
- Drink warm, sweet beverages (sugar water, sports-type drinks.) **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Eat warm, high-calorie foods like hot pasta dishes.

Workers are at increased risk when…

- They have predisposing health conditions such as cardiovascular disease, diabetes, and hypertension.
- They take certain medications. Check with your doctor, nurse, or pharmacy and ask if medicines you take affect you while working in cold environments.
- They are in poor physical condition, have a poor diet, or are older.

HYPOTHERMIA - (Medical Emergency)

What happens to the body:

Normal body temperature (98.6°F/37°C) drops to or below 95°F/35°C; fatigue or drowsiness; uncontrolled shivering; cool, bluish skin; slurred speech; clumsy movements; irritable, irrational, or confused behavior.

What to do: (land temperatures)

- Call for emergency help (i.e., ambulance or 911).
- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet clothing and replace with warm, dry clothing or wrap the person in blankets.
- Have the person drink warm, sweet drinks (sugar water or sports-type drinks) if he is alert. **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Have the person move his arms and legs to create muscle heat. If he is unable to do this, place warm bottles or hot packs in the armpits, groin, neck, and head areas. **Do not** rub the person's body or place him in a warm water bath. This may stop his heart.

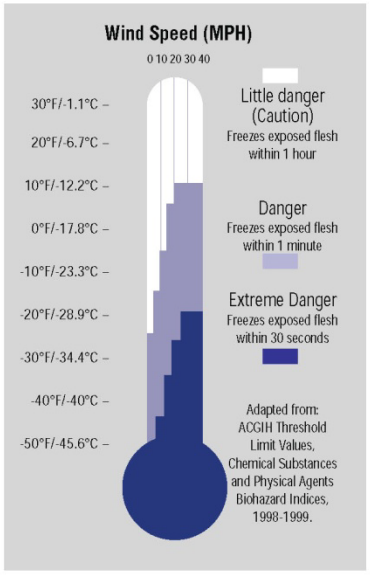
What to do: (water temperatures)

- Call for emergency help (i.e., ambulance or 911). Body heat is lost up to 25 times faster in water.
- **Do not** remove any clothing. Button, buckle, zip, and tighten any collars, cuffs, shoes, and hoods because the layer of trapped water closest to the body provides a layer of insulation that slows the loss of heat. Keep the head out of the water and put on a hat or hood.
- Get out of the water as quickly as possible or climb on anything floating. **Do not** attempt to swim unless a floating object or another person can be reached because swimming or other physical activity uses body heat and reduces survival time by about 50 percent.
- If getting out of the water is not possible, wait quietly and conserve body heat by folding arms across the chest, keeping thighs together, bending knees, and crossing ankles. If another person is in the water, huddle together with chests held closely.

THE COLD STRESS EQUATION

LOW TEMPERATURE + WIND SPEED + WETNESS = INJURIES & ILLNESS

When the body is unable to warm itself, serious cold-related illnesses and injuries may occur, and permanent tissue damage and death may result. **Hypothermia** can occur when *land temperatures* are **above** freezing or *water temperatures* are below 98.6°F/37°C. Cold-related illnesses can slowly overcome a person who has been chilled by low temperatures, brisk winds, or wet clothing.



Oregon Occupational Safety & Health Division
440-3336E S (9/03)

HEAT EXHAUSTION

What happens to the body:

Headaches, dizziness, or light-headedness, weakness, mood changes, irritability or confusion, feeling sick to your stomach, vomiting, fainting, decreased and dark-colored urine, and pale, clammy skin.

What should be done:

- Move the person to a cool shaded area. Don't leave the person alone. If the person is dizzy or light-headed, lay him on his back and raise his legs about 6-8 inches. If the person is sick to his stomach, lay him on his side.
- Loosen and remove heavy clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is not feeling sick to his stomach.
- Try to cool the person by fanning him. Cool the skin with a cool spray mist of water or wet cloth.
- If the person does not feel better in a few minutes call for emergency help (ambulance or call 911.)

(If heat exhaustion is not treated, the illness may advance to heat stroke.)

HEAT STROKE - A Medical Emergency

What happens to the body:

Dry, pale skin (no sweating); hot red skin (looks like a sunburn); mood changes; irritability, confusion, and not making any sense; seizures or fits, and collapse (will not respond).

What should be done:

- Call for emergency help (i.e., ambulance or 911.)
- Move the person to a cool, shaded area. Don't leave the person alone. Lay him on his back and if the person is having seizures, remove objects close to him so he won't hit them. If the person is sick to his stomach, lay him on his side.
- Remove heavy and outer clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is alert enough to drink anything and not feeling sick to his stomach.
- Try to cool the person by fanning him or her. Cool the skin with a cool spray mist of water, wet cloth, or wet sheet.
- If ice is available, place ice packs in armpits and groin area.

How to Protect Workers

- Learn the signs and symptoms of heat-induced illnesses and what to do to help the worker.
- Train workers about heat-induced illnesses.
- Perform the heaviest work during the coolest part of the day.
- Slowly build up tolerance to the heat and the work activity (usually takes up to 2 weeks.)
- Use the buddy system (work in pairs.)
- Drink plenty of cool water (one small cup every 15-20 minutes.)
- Wear light, loose-fitting, breathable (like cotton) clothing.
- Take frequent short breaks in cool, shaded areas (allow your body to cool down.)
- Avoid eating large meals before working in hot environments.
- Avoid caffeine and alcoholic beverages (these beverages make the body lose water and increase the risk of heat illnesses.)

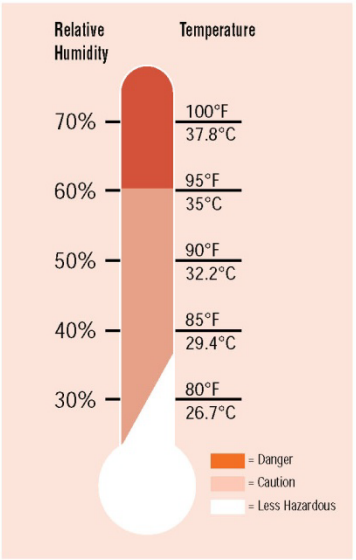
Workers are at increased risk when...

- They take certain medications. Check with your doctor, nurse, or pharmacy to see if medicines you take affect you when working in hot environments.
- They have had a heat-induced illness in the past.
- They wear personal protective equipment.

THE HEAT EQUATION

HIGH TEMPERATURE + HIGH HUMIDITY + PHYSICAL WORK = HEAT ILLNESS

When the body is unable to cool itself through sweating, **serious** heat illnesses may occur. The most severe heat-induced illnesses are **heat exhaustion** and **heat stroke**. If actions are not taken to treat heat exhaustion, the illness could progress to heat stroke and **death**.



Oregon Occupational Safety & Health Division
440-3333E-S(9/03)

ATTACHMENT 4

SAFETY DATA SHEETS

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

1 Identification of the substance/mixture and of the supplier

1.1 Product identifier

Trade Name: Alconox
Synonyms:
Product number: 1104-1, 1104, 1125, 1150, 1101, 1103, 1112-1, 1112

1.2 Application of the substance / the mixture : Cleaning material/Detergent

1.3 Details of the supplier of the Safety Data Sheet

Manufacturer
Alconox, Inc.
30 Glenn Street
White Plains, NY 10603
1-914-948-4040

Supplier

Emergency telephone number:
ChemTel Inc
North America: 1-800-255-3924
International: 01-813-248-0585

2 Hazards identification

2.1 Classification of the substance or mixture:

In compliance with EC regulation No. 1272/2008, 29CFR1910/1200 and GHS Rev. 3 and amendments.

Hazard-determining components of labeling:
Tetrasodium Pyrophosphate
Sodium tripolyphosphate
Sodium Alkylbenzene Sulfonate

2.2 Label elements:

Skin irritation, category 2.
Eye irritation, category 2A.

Hazard pictograms:

Signal word: Warning

Hazard statements:
H315 Causes skin irritation.
H319 Causes serious eye irritation.

Precautionary statements:
P264 Wash skin thoroughly after handling.
P280 Wear protective gloves/protective clothing/eye protection/face protection.
P302+P352 If on skin: Wash with soap and water.
P305+P351+P338 If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.
P321 Specific treatment (see supplemental first aid instructions on this label).
P332+P313 If skin irritation occurs: Get medical advice/attention.
P362 Take off contaminated clothing and wash before reuse.
P501 Dispose of contents and container as instructed in Section 13.

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

Additional information: None.

Hazard description

Hazards Not Otherwise Classified (HNOC): None

Information concerning particular hazards for humans and environment:

The product has to be labelled due to the calculation procedure of the "General Classification guideline for preparations of the EU" in the latest valid version.

Classification system:

The classification is according to EC regulation No. 1272/2008, 29CFR1910/1200 and GHS Rev. 3 and amendments, and extended by company and literature data. The classification is in accordance with the latest editions of international substances lists, and is supplemented by information from technical literature and by information provided by the company.

3 Composition/information on ingredients

3.1 Chemical characterization : None

3.2 Description : None

3.3 Hazardous components (percentages by weight)

Identification	Chemical Name	Classification	Wt. %
CAS number: 7758-29-4	Sodium tripolyphosphate	Skin Irrit. 2 ; H315 Eye Irrit. 2; H319	12-28
CAS number: 68081-81-2	Sodium Alkylbenzene Sulfonate	Acute Tox. 4; H303 Skin Irrit. 2 ; H315 Eye Irrit. 2; H319	8-22
CAS number: 7722-88-5	Tetrasodium Pyrophosphate	Skin Irrit. 2 ; H315 Eye Irrit. 2; H319	2-16

3.4 Additional Information : None.

4 First aid measures

4.1 Description of first aid measures

General information: None.

After inhalation:

Maintain an unobstructed airway.

Loosen clothing as necessary and position individual in a comfortable position.

After skin contact:

Wash affected area with soap and water.

Seek medical attention if symptoms develop or persist.

After eye contact:

Rinse/flush exposed eye(s) gently using water for 15-20 minutes.

Remove contact lens(es) if able to do so during rinsing.

Seek medical attention if irritation persists or if concerned.

After swallowing:

Rinse mouth thoroughly.

Seek medical attention if irritation, discomfort, or vomiting persists.

4.2 Most important symptoms and effects, both acute and delayed

None

4.3 Indication of any immediate medical attention and special treatment needed:

No additional information.

5 Firefighting measures

5.1 Extinguishing media

Suitable extinguishing agents:

Use appropriate fire suppression agents for adjacent combustible materials or sources of ignition.

For safety reasons unsuitable extinguishing agents : None

5.2 Special hazards arising from the substance or mixture :

Thermal decomposition can lead to release of irritating gases and vapors.

5.3 Advice for firefighters

Protective equipment:

Wear protective eye wear, gloves and clothing.
Refer to Section 8.

5.4 Additional information :

Avoid inhaling gases, fumes, dust, mist, vapor and aerosols.
Avoid contact with skin, eyes and clothing.

6 Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures :

Ensure adequate ventilation.
Ensure air handling systems are operational.

6.2 Environmental precautions :

Should not be released into the environment.
Prevent from reaching drains, sewer or waterway.

6.3 Methods and material for containment and cleaning up :

Wear protective eye wear, gloves and clothing.

6.4 Reference to other sections : None

7 Handling and storage

7.1 Precautions for safe handling :

Avoid breathing mist or vapor.
Do not eat, drink, smoke or use personal products when handling chemical substances.

7.2 Conditions for safe storage, including any incompatibilities :

Store in a cool, well-ventilated area.

7.3 Specific end use(s):

No additional information.

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

8 Exposure controls/personal protection

8.1 Control parameters :

a) 7722-88-5, Tetrasodium Pyrophosphate, OSHA TWA 5 mg/m3

b) Dusts, non-specific OEL, Irish Code of Practice

(i) Total inhalable 10 mg/m3 (8hr)

(ii) Respirible 4mg/m3 (8hr)

(iii) Tetrasodium Pyrophosphate, OSHA TWA 5 mg/m3, (8hr)

8.2 Exposure controls

Appropriate engineering controls:

Emergency eye wash fountains and safety showers should be available in the immediate vicinity of use or handling.

Respiratory protection:

Not needed under normal use conditions.

Protection of skin:

Select glove material impermeable and resistant to the substance or preparation. Protective gloves recommended to comply with EN 374. Take note of break through times, permeability, and special workplace conditions, such as mechanical strain, duration of contact, etc. Protective gloves should be replaced at the first sign of wear.

Eye protection:

Safety goggles or glasses, or appropriate eye protection. Recommended to comply with ANSI Z87.1 and/or EN 166.

General hygienic measures:

Wash hands before breaks and at the end of work.

Avoid contact with skin, eyes and clothing.

9 Physical and chemical properties

Appearance (physical state, color):	White and cream colored flakes - powder	**Explosion limit lower:** **Explosion limit upper:**	Not determined or not available. Not determined or not available.
Odor:	Not determined or not available.	**Vapor pressure at 20°C:**	Not determined or not available.
Odor threshold:	Not determined or not available.	**Vapor density:**	Not determined or not available.
pH-value:	9.5 (aqueous solution)	**Relative density:**	Not determined or not available.
Melting/Freezing point:	Not determined or not available.	**Solubilities:**	Not determined or not available.
Boiling point/Boiling range:	Not determined or not available.	**Partition coefficient (n-octanol/water):**	Not determined or not available.
Flash point (closed cup):	Not determined or not available.	**Auto/Self-ignition temperature:**	Not determined or not available.
Evaporation rate:	Not determined or not available.	**Decompositio n**	Not determined or not available.

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

Flammability (solid, gaseous):	Not determined or not available.	**Viscosity**:	a. Kinematic: Not determined or not available. b. Dynamic: Not determined or not available.
Density at 20°C:	Not determined or not available.		

10 Stability and reactivity

10.1 Reactivity : None

10.2 Chemical stability : None

10.3 Possibility hazardous reactions : None

10.4 Conditions to avoid : None

10.5 Incompatible materials : None

10.6 Hazardous decomposition products : None

11 Toxicological information

11.1 Information on toxicological effects :

Acute Toxicity:

Oral:

: LD50 > 5000 mg/kg oral rat - Product .

Chronic Toxicity: No additional information.

Skin corrosion/irritation:

Sodium Alkylbenzene Sulfonate: Causes skin irritation. .

Serious eye damage/irritation:

Sodium Alkylbenzene Sulfonate: Causes serious eye irritation .

Tetrasodium Pyrophosphate: Rabbit - Risk of serious damage to eyes .

Respiratory or skin sensitization: No additional information.

Carcinogenicity: No additional information.

IARC (International Agency for Research on Cancer): None of the ingredients are listed.

NTP (National Toxicology Program): None of the ingredients are listed.

Germ cell mutagenicity: No additional information.

Reproductive toxicity: No additional information.

STOT-single and repeated exposure: No additional information.

Additional toxicological information: No additional information.

12 Ecological information

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

12.1 Toxicity:

Sodium Alkylbenzene Sulfonate: Fish, LC50 1.67 mg/l, 96 hours.
Sodium Alkylbenzene Sulfonate: Aquatic invertebrates, EC50 Daphnia 2.4 mg/l, 48 hours. Sodium Alkylbenzene Sulfonate: Aquatic Plants, EC50 Algae 29 mg/l, 96 hours.
Tetrasodium Pyrophosphate: Fish, LC50 - other fish - 1,380 mg/l - 96 h.
Tetrasodium Pyrophosphate: Aquatic invertebrates, EC50 - Daphnia magna (Water flea) - 391 mg/l - 48 h.

12.2 Persistence and degradability: No additional information.

12.3 Bioaccumulative potential: No additional information.

12.4 Mobility in soil: No additional information.

General notes: No additional information.

12.5 Results of PBT and vPvB assessment:

PBT: No additional information.

vPvB: No additional information.

12.6 Other adverse effects: No additional information.

13 Disposal considerations

13.1 Waste treatment methods (consult local, regional and national authorities for proper disposal)

Relevant Information:

It is the responsibility of the waste generator to properly characterize all waste materials according to applicable regulatory entities. (US 40CFR262.11).

14 Transport information

14.1 UN Number: ADR, ADN, DOT, IMDG, IATA	None
14.2 UN Proper shipping name: ADR, ADN, DOT, IMDG, IATA	None
14.3 Transport hazard classes: ADR, ADN, DOT, IMDG, IATA	**Class:** None **Label:** None **LTD.QTY:** None

US DOT

Limited Quantity Exception: None

Bulk:
RQ (if applicable): None
Proper shipping Name: None
Hazard Class: None
Packing Group: None
Marine Pollutant (if applicable): No additional information.

Non Bulk:
RQ (if applicable): None
Proper shipping Name: None
Hazard Class: None
Packing Group: None
Marine Pollutant (if applicable): No additional information.

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

Comments: None	**Comments:** None
14.4 Packing group: ADR, ADN, DOT, IMDG, IATA	None
14.5 Environmental hazards :	None
14.6 Special precautions for user:	None
Danger code (Kemler):	None
EMS number:	None
Segregation groups:	None
14.7 Transport in bulk according to Annex II of MARPOL73/78 and the IBC Code: Not applicable.	
14.8 Transport/Additional information:	
Transport category:	None
Tunnel restriction code:	None
UN "Model Regulation":	None

15 Regulatory information

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture.

North American

SARA

Section 313 (specific toxic chemical listings): None of the ingredients are listed.
Section 302 (extremely hazardous substances): None of the ingredients are listed.

CERCLA (Comprehensive Environmental Response, Clean up and Liability Act) Reportable

Spill Quantity: None of the ingredients are listed.

TSCA (Toxic Substances Control Act):

Inventory: All ingredients are listed.
Rules and Orders: Not applicable.

Proposition 65 (California):

Chemicals known to cause cancer: None of the ingredients are listed.

Chemicals known to cause reproductive toxicity for females: None of the ingredients are listed.

Chemicals known to cause reproductive toxicity for males: None of the ingredients are listed.

Chemicals known to cause developmental toxicity: None of the ingredients are listed.

Canadian

Canadian Domestic Substances List (DSL):
All ingredients are listed.

EU

REACH Article 57 (SVHC): None of the ingredients are listed.

Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

Effective date: 10.18.2017 **Revision**: 10.18.2017

Trade Name: Alconox

Germany MAK: Not classified.
EC 648/2004 – This is an industrial detergent. Contains >30% phosphate, 15-30% anionic surfactant, <5% EDTA salts
EC 551/2009 – This is not a laundry or dishwasher detergent
EC 907/2006 – Contains no enzymes, optical brighteners, perfumes, allergenic fragrances, or preservative agents

Asia Pacific

Australia

Australian Inventory of Chemical Substances (AICS): All ingredients are listed.

China

Inventory of Existing Chemical Substances in China (IECSC): All ingredients are listed.

Japan

Inventory of Existing and New Chemical Substances (ENCS): All ingredients are listed.

Korea

Existing Chemicals List (ECL): All ingredients are listed.

New Zealand

New Zealand Inventory of Chemicals (NZOIC): All ingredients are listed.

Philippines

Philippine Inventory of Chemicals and Chemical Substances (PICCS): All ingredients are listed.

Taiwan

Taiwan Chemical Substance Inventory (TSCI): All ingredients are listed.

16 Other information

Abbreviations and Acronyms: None

Summary of Phrases

Hazard statements:
H315 Causes skin irritation.
H319 Causes serious eye irritation.

NFPA: 1-0-0
HMIS: 1-0-0

Precautionary statements:
P264 Wash skin thoroughly after handling.
P280 Wear protective gloves/protective clothing/eye protection/face protection.
P302+P352 If on skin: Wash with soap and water.
P305+P351+P338 If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.
P321 Specific treatment (see supplemental first aid instructions on this label).
P332+P313 If skin irritation occurs: Get medical advice/attention.
P362 Take off contaminated clothing and wash before reuse.
P501 Dispose of contents and container as instructed in Section 13.

Manufacturer Statement:

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

SAFETY DATA SHEET

according to the (US) Hazard Communication Standard (29 CFR 1910.1200)

Revision Date 01/27/2015 Version 1.2

SECTION 1.Identification

Product identifier

Product number	HX0607
Product name	Hydrochloric Acid 34-37% OmniTrace®

Relevant identified uses of the substance or mixture and uses advised against

Identified uses	Reagent for research and development

Details of the supplier of the safety data sheet

Company	EMD Millipore Corporation | 290 Concord Road, Billerica, MA 01821, United States of America | General Inquiries: +1-978-715-4321 | Monday to Friday, 9:00 AM to 4:00 PM Eastern Time (GMT-5)
Emergency telephone	800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2. Hazards identification

GHS Classification

Corrosive to Metals, Category 1, H290
Skin corrosion, Category 1B, H314
Serious eye damage, Category 1, H318
Specific target organ systemic toxicity - single exposure, Category 3, Respiratory system, H335

For the full text of the H-Statements mentioned in this Section, see Section 16.

GHS-Labeling

Hazard pictograms

Signal Word
Danger

Hazard Statements
H290 May be corrosive to metals.
H314 Causes severe skin burns and eye damage.
H335 May cause respiratory irritation.

Precautionary Statements
P234 Keep only in original container.
P261 Avoid breathing dust/ fume/ gas/ mist/ vapors/ spray.
P264 Wash skin thoroughly after handling.
P271 Use only outdoors or in a well-ventilated area.
P280 Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
P303 + P361 + P353 IF ON SKIN (or hair): Remove/ Take off immediately all contaminated clothing. Rinse skin with water/ shower.
P304 + P340 IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P310 Immediately call a POISON CENTER or doctor/ physician.
P321 Specific treatment (see supplemental first aid instructions on this label).
P363 Wash contaminated clothing before reuse.
P390 Absorb spillage to prevent material damage.
P403 + P233 Store in a well-ventilated place. Keep container tightly closed.
P405 Store locked up.
P406 Store in corrosive resistant stainless steel container with a resistant inliner.
P501 Dispose of contents/ container to an approved waste disposal plant.

Other hazards

None known.

SECTION 3. Composition/information on ingredients

Chemical nature Aqueous solution

Hazardous ingredients

Chemical Name (Concentration)
CAS-No.
hydrochloric acid (>= 30 % - < 50 %)
7647-01-0

Exact percentages are being wihtheld as a trade secret.

SECTION 4. First aid measures

Description of first-aid measures

General advice
First aider needs to protect himself.

Inhalation
After inhalation: fresh air. Call in physician.

Skin contact
In case of skin contact: Take off immediately all contaminated clothing. Rinse skin with water/ shower. Call a physician immediately.

Eye contact
After eye contact: rinse out with plenty of water. Immediately call in ophthalmologist.

Ingestion
After swallowing: make victim drink water (two glasses at most), avoid vomiting (risk of perforation!). Call a physician immediately. Do not attempt to neutralize.

Never give anything by mouth to an unconscious person.

Most important symptoms and effects, both acute and delayed
Irritation and corrosion, Cough, Shortness of breath, cardiovascular disorders, Risk of blindness!

Indication of any immediate medical attention and special treatment needed
No information available.

SECTION 5. Fire-fighting measures

Extinguishing media

Suitable extinguishing media
Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Unsuitable extinguishing media
For this substance/mixture no limitations of extinguishing agents are given.

Special hazards arising from the substance or mixture
Not combustible.
Ambient fire may liberate hazardous vapors.
Fire may cause evolution of:
Hydrogen chloride gas

Advice for firefighters

Special protective equipment for fire-fighters
Stay in danger area only with self-contained breathing apparatus. Prevent skin contact by keeping a safe distance or by wearing suitable protective clothing.

Further information
Suppress (knock down) gases/vapors/mists with a water spray jet. Prevent fire extinguishing water from contaminating surface water or the ground water system.

SECTION 6. Accidental release measures

Personal precautions, protective equipment and emergency procedures
Advice for non-emergency personnel: Do not breathe vapors, aerosols. Avoid substance contact. Ensure adequate ventilation. Evacuate the danger area, observe emergency procedures, consult an expert.

Advice for emergency responders: Protective equipment see section 8.

Environmental precautions
Do not empty into drains.

Methods and materials for containment and cleaning up
Cover drains. Collect, bind, and pump off spills.
Observe possible material restrictions (see sections 7 and 10).
Take up with liquid-absorbent and neutralizing material (e.g. Chemizorb® H^+, Art. No. 101595).
Dispose of properly. Clean up affected area.

SECTION 7. Handling and storage

Precautions for safe handling

Observe label precautions.

Conditions for safe storage, including any incompatibilities

Requirements for storage areas and containers

No metal containers.

Tightly closed.

Store at room temperature.

SECTION 8. Exposure controls/personal protection

Exposure limit(s)

Ingredients

Basis	Value	Threshold limits	Remarks
hydrochloric acid 7647-01-0			
ACGIH	Ceiling Limit Value:	2 ppm	
NIOSH/GUIDE	Ceiling Limit Value and Time Period (if specified):	5 ppm 7 mg/m³	
OSHA_TRANS	Ceiling Limit Value:	5 ppm 7 mg/m³	
Z1A	Ceiling Limit Value:	5 ppm 7 mg/m³	

Engineering measures

Technical measures and appropriate working operations should be given priority over the use of personal protective equipment.

Individual protection measures

Protective clothing should be selected specifically for the workplace, depending on concentration and quantity of the hazardous substances handled. The chemical resistance of the protective equipment should be inquired at the respective supplier.

Hygiene measures

Immediately change contaminated clothing. Apply skin- protective barrier cream. Wash hands and face after working with substance.

Eye/face protection

Tightly fitting safety goggles

Hand protection

Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary.

Other protective equipment:

Acid-resistant protective clothing.

Respiratory protection
required when vapors/aerosols are generated.
Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.

SECTION 9. Physical and chemical properties

Physical state	liquid
Color	colorless
Odor	stinging
Odor Threshold	0.8 - 5 ppm Gaseous hydrogen chloride (HCl).
pH	< 1 at 68 °F (20 °C)
Solidification point	-30 °C
Boiling point	No information available.
Flash point	Not applicable
Evaporation rate	No information available.
Flammability (solid, gas)	No information available.
Lower explosion limit	Not applicable
Upper explosion limit	Not applicable
Vapor pressure	190 hPa at 68 °F (20 °C)
Relative vapor density	No information available.
Density	ca.1.19 g/cm³ at 68 °F (20 °C)
Relative density	No information available.
Water solubility	at 68 °F (20 °C) soluble
Partition coefficient: n-octanol/water	Not applicable

Autoignition temperature	No information available.
Decomposition temperature	No information available.
Viscosity, dynamic	2.3 mPa.s at 59 °F (15 °C)
Explosive properties	Not classified as explosive.
Oxidizing properties	none
Ignition temperature	Not applicable
Corrosion	May be corrosive to metals.

SECTION 10. Stability and reactivity

Reactivity

Corrosive in contact with metals

Chemical stability

The product is chemically stable under standard ambient conditions (room temperature) .

Possibility of hazardous reactions

Exothermic reaction with:

Amines, potassium permanganate, salts of oxyhalogenic acids, semimetallic oxides, semimetallic hydrogen compounds, Aldehydes, vinylmethyl ether

Risk of ignition or formation of inflammable gases or vapors with:

carbides, lithium silicide, Fluorine

Generates dangerous gases or fumes in contact with:

Aluminum, hydrides, formaldehyde, Metals, strong alkalis, Sulfides

Risk of explosion with:

Alkali metals, conc. sulfuric acid

Conditions to avoid

Heating.

Incompatible materials

Metals, metal alloys
Gives off hydrogen by reaction with metals.

Hazardous decomposition products

in the event of fire: See section 5.

SECTION 11. Toxicological information

Information on toxicological effects

Likely route of exposure
Inhalation, Eye contact, Skin contact

Target Organs
Eyes
Skin
Respiratory system
Cornea

Acute oral toxicity
Symptoms: If ingested, severe burns of the mouth and throat, as well as a danger of perforation of the esophagus and the stomach.

Acute toxicity estimate: 1,892 mg/kg
Calculation method

Acute inhalation toxicity

Symptoms: mucosal irritations, Cough, Shortness of breath, Possible damages:, damage of respiratory tract

Acute toxicity estimate: 6.41 mg/l; 4 h
Calculation method

Skin irritation
Mixture causes burns.

Eye irritation
Mixture causes serious eye damage. Risk of blindness!

Specific target organ systemic toxicity - single exposure
Target Organs: Respiratory system
Mixture may cause respiratory irritation.

Specific target organ systemic toxicity - repeated exposure
The substance or mixture is not classified as specific target organ toxicant, repeated exposure.

Aspiration hazard
Regarding the available data the classification criteria are not fulfilled.

Carcinogenicity

IARC	No ingredient of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
OSHA	No ingredient of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.
NTP	No ingredient of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
ACGIH	No ingredient of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by ACGIH.

Further information

After uptake:
After a latency period:
cardiovascular disorders
Handle in accordance with good industrial hygiene and safety practice.

Ingredients

hydrochloric acid
No information available.

SECTION 12. Ecological information

Ecotoxicity

No information available.

Persistence and degradability

No information available.

Bioaccumulative potential

Partition coefficient: n-octanol/water
Not applicable

Mobility in soil

No information available.

Additional ecological information
Forms corrosive mixtures with water even if diluted. Harmful effect due to pH shift.
Discharge into the environment must be avoided.

Ingredients

hydrochloric acid

Substance does not meets the criteria for PBT or vPvB according to Regulation (EC) No 1907/2006, Annex XIII.

SECTION 13. Disposal considerations

The information presented only applies to the material as supplied. The identification based on characteristic(s) or listing may not apply if the material has been used or otherwise contaminated. It is the responsibility of the waste generator to determine the toxicity and physical properties of the material generated to determine the proper waste identification and disposal methods in compliance with applicable regulations. Disposal should be in accordance with applicable regional, national and local laws and regulations.

SECTION 14. Transport information

Land transport (DOT)

UN number	UN 1789
Proper shipping name	HYDROCHLORIC ACID
Class	8
Packing group	II
Environmentally hazardous	--

Air transport (IATA)

UN number	UN 1789
Proper shipping name	HYDROCHLORIC ACID
Class	8
Packing group	II
Environmentally hazardous	--
Special precautions for user	no

Sea transport (IMDG)

UN number	UN 1789
Proper shipping name	HYDROCHLORIC ACID
Class	8
Packing group	II
Environmentally hazardous	--
Special precautions for user	yes
EmS	F-A S-B

SECTION 15. Regulatory information

United States of America

SARA 313

The following components are subject to reporting levels established by SARA Title III, Section 313:

Ingredients

hydrochloric acid	7647-01-0	*37 %*

SARA 302

The following components are subject to reporting levels established by SARA Title III, Section 302:

Ingredients

hydrochloric acid	7647-01-0

Clean Water Act

The following Hazardous Substances are listed under the U.S. CleanWater Act, Section 311, Table 116.4A:
Ingredients
hydrochloric acid

The following Hazardous Chemicals are listed under the U.S. CleanWater Act, Section 311, Table 117.3:
Ingredients
hydrochloric acid

DEA List I
Not listed

DEA List II
Listed
Ingredients

hydrochloric acid	7647-01-0

US State Regulations

Massachusetts Right To Know
Ingredients
hydrochloric acid

Pennsylvania Right To Know
Ingredients
hydrochloric acid

New Jersey Right To Know
Ingredients
hydrochloric acid

California Prop 65 Components
This product does not contain any chemicals known to the State of California to cause cancer, birth, or any other reproductive defects.

Notification status

TSCA:	All components of the product are listed in the TSCA-inventory.
DSL:	All components of this product are on the Canadian DSL.
KOREA:	Not in compliance with the inventory

SECTION 16. Other information

Training advice
Provide adequate information, instruction and training for operators.

Labeling

Hazard pictograms

Signal Word
Danger

Hazard Statements
H290 May be corrosive to metals.
H314 Causes severe skin burns and eye damage.
H335 May cause respiratory irritation.

Precautionary Statements
Prevention
P280 Wear protective gloves/ protective clothing/ eye protection/ face protection.
Response
P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P308 + P310 IF exposed or concerned: immediately call a POISON CENTER or doctor/ physician.

Full text of H-Statements referred to under sections 2 and 3.

H290	May be corrosive to metals.
H314	Causes severe skin burns and eye damage.
H318	Causes serious eye damage.
H335	May cause respiratory irritation.

Key or legend to abbreviations and acronyms used in the safety data sheet
Used abbreviations and acronyms can be looked up at www.wikipedia.org.

Revision Date01/27/2015

Isobutylene (0.0005% - 1.34%), Oxygen (19.5 - 23.5%) in balance Nitrogen

Safety Data Sheet 50054

according to Federal Register / Vol. 77, No. 58 / Monday, March 26, 2012 / Rules and Regulations

Date of issue: 03/24/2015 Revision date: 03/01/2018 Supersedes: 03/24/2015 Version: 1.1

SECTION 1: Identification

1.1. Identification

Product form : Mixtures

Product name : Isobutylene (0.0005% - 1.34%), Oxygen (19.5 - 23.5%) in balance Nitrogen

1.2. Recommended use and restrictions on use

Use of the substance/mixture : Test gas/Calibration gas.

1.3. Supplier

Calgaz, division of Airgas USA LLC
821 Chesapeake Drive
Cambridge, 21613 - USA
T 1-410-228-6400 - F 1-410-228-4251
info@Calgaz.com - www.Calgaz.com

1.4. Emergency telephone number

Emergency number : CHEMTREC: 1-800-424-9300
Internationally: 1-703-527-3887

SECTION 2: Hazard(s) identification

2.1. Classification of the substance or mixture

GHS-US classification

Gases under pressure Compressed gas H280 Contains gas under pressure; may explode if heated

Full text of H statements : see section 16

2.2. GHS Label elements, including precautionary statements

GHS-US labeling

Hazard pictograms (GHS-US) :

GHS04

Signal word (GHS-US) : Warning

Hazard statements (GHS-US) : H280 - Contains gas under pressure; may explode if heated

Precautionary statements (GHS-US) : P202 - Do not handle until all safety precautions have been read and understood.
P271 - Use only outdoors or in a well-ventilated area.
P403 - Store in a well-ventilated place.
CGA-PG02 - Protect from sunlight when ambient temperature exceeds 52°C/125 °F
CGA-PG05 - Use a back flow preventive device in the piping
CGA-PG06 - Close valve after each use and when empty
CGA-PG10 - Use only with equipment rated for cylinder pressure
CGA-PG14 - Approach suspected leak area with caution
CGA-PG21 - Open valve slowly

2.3. Other hazards which do not result in classification

No additional information available

2.4. Unknown acute toxicity (GHS US)

Not applicable

SECTION 3: Composition/Information on ingredients

3.1. Substances

Not applicable

3.2. Mixtures

Name	Product identifier	%	GHS-US classification
Nitrogen	(CAS-No.) 7727-37-9	75.16 - 80.4995	Press. Gas (Comp.), H280
Oxygen	(CAS-No.) 7782-44-7	19.5 - 23.5	Ox. Gas 1, H270 Press. Gas (Comp.), H280
Isobutylene	(CAS-No.) 115-11-7	0.0005 - 1.34	Press. Gas (Liq.), H280

Full text of hazard classes and H-statements : see section 16

SECTION 4: First-aid measures

4.1. Description of first aid measures

First-aid measures general : Adverse effects not expected from this product. If you feel unwell, seek medical advice (show the label where possible).

First-aid measures after inhalation : Adverse effects not expected from this product.

First-aid measures after skin contact : Adverse effects not expected from this product.

First-aid measures after eye contact : Adverse effects not expected from this product.

First-aid measures after ingestion : Ingestion is not considered a potential route of exposure.

4.2. Most important symptoms and effects (acute and delayed)

Symptoms/effects after inhalation : Adverse effects not expected from this product.

Symptoms/effects after skin contact : Adverse effects not expected from this product.

Symptoms/effects after eye contact : Adverse effects not expected from this product.

Symptoms/effects after ingestion : Ingestion is not considered a potential route of exposure.

Symptoms/effects upon intravenous administration : Not known.

Chronic symptoms : Adverse effects not expected from this product.

Most important symptoms and effects, both acute and delayed : No effect on living tissue. Refer to section 11.

4.3. Immediate medical attention and special treatment, if necessary

If you feel unwell, seek medical advice. If breathing is difficult, give oxygen.

SECTION 5: Fire-fighting measures

5.1. Suitable (and unsuitable) extinguishing media

Suitable extinguishing media : Use extinguishing media appropriate for surrounding fire.

Unsuitable extinguishing media : Do not use water jet to extinguish.

5.2. Specific hazards arising from the chemical

Fire hazard : The product is not flammable.

Explosion hazard : Product is not explosive. Heat may build pressure, rupturing closed containers, spreading fire and increasing risk of burns and injuries.

Reactivity : None known.

Hazardous combustion products : None known

5.3. Special protective equipment and precautions for fire-fighters

Firefighting instructions : In case of fire: Evacuate area. Fight fire remotely due to the risk of explosion. Use water spray or fog for cooling exposed containers. Exercise caution when fighting any chemical fire.

Protection during firefighting : Standard protective clothing and equipment (e.g, Self Contained Breathing Apparatus) for fire fighters. Do not enter fire area without proper protective equipment, including respiratory protection.

Specific methods : Exposure to fire may cause containers to rupture/explode. Continue water spray from protected position until container stays cool. Move containers away from the fire area if this can be done without risk.

SECTION 6: Accidental release measures

6.1. Personal precautions, protective equipment and emergency procedures

General measures : Ensure adequate ventilation.

6.1.1. For non-emergency personnel

Protective equipment : Wear protective equipment consistent with the site emergency plan.

Emergency procedures	:	Evacuate personnel to a safe area. Close doors and windows of adjacent premises. Keep containers closed. Mark the danger area. Seal off low-lying areas. Keep upwind.

6.1.2. For emergency responders

Protective equipment	:	Standard protective clothing and equipment (e.g, Self Contained Breathing Apparatus) for fire fighters. Equip cleanup crew with proper protection.
Emergency procedures	:	Evacuate and limit access. Ventilate area.

6.2. Environmental precautions

Try to stop release if without risk.

6.3. Methods and material for containment and cleaning up

For containment	:	Try to stop release if without risk.
Methods for cleaning up	:	Dispose of contents/container in accordance with local/regional/national/international regulations.
Methods and material for containment and cleaning up	:	None.

6.4. Reference to other sections

See also Sections 8 and 13.

SECTION 7: Handling and storage

7.1. Precautions for safe handling

Additional hazards when processed	:	Pressurized container: Do not pierce or burn, even after use. Use only with equipment rated for cylinder pressure.
Precautions for safe handling	:	Do not handle until all safety precautions have been read and understood. Use only outdoors or in a well-ventilated area.
Safe handling of the gas receptacle	:	Protect cylinders from physical damage; do not drag, roll, slide or drop. Do not remove or deface labels provided by the supplier for the identification of the cylinder contents.
Safe use of the product	:	The product must be handled in accordance with good industrial hygiene and safety procedures. Only experienced and properly instructed persons should handle gases under pressure. Consider pressure relief device(s) in gas installations. Ensure the complete gas system was (or is regularily) checked for leaks before use. Do not remove or deface labels provided by the supplier for the identification of the cylinder contents. Use only properly specified equipment which is suitable for this product, its supply pressure and temperature. Contact your gas supplier if in doubt.
Hygiene measures	:	Do not eat, drink or smoke when using this product.

7.2. Conditions for safe storage, including any incompatibilities

Technical measures	:	None known.
Storage conditions	:	Do not expose to temperatures exceeding 52 °C/ 125 °F. Keep container closed when not in use. Protect cylinders from physical damage; do not drag, roll, slide or drop. Store in well ventilated area.
Incompatible products	:	None known.
Incompatible materials	:	Flammable materials.
Conditions for safe storage, including any incompatibilities	:	Observe all regulations and local requirements regarding storage of containers. Containers should not be stored in conditions likely to encourage corrosion. Container valve guards or caps should be in place. Containers should be stored in the vertical position and properly secured to prevent them from falling over. Stored containers should be periodically checked for general condition and leakage. Keep container below 50°C in a well ventilated place. Store containers in location free from fire risk and away from sources of heat and ignition. Keep away from combustible materials.
Storage area	:	Store away from heat. Store in a well-ventilated place.

SECTION 8: Exposure controls/personal protection

8.1. Control parameters

Isobutylene (115-11-7)		
ACGIH	ACGIH TWA (ppm)	250 ppm

Oxygen (7782-44-7)
Not applicable

Nitrogen (7727-37-9)		
ACGIH	Remark (ACGIH)	Simple Asphyxiant

8.2. Appropriate engineering controls

Appropriate engineering controls : Provide adequate general and local exhaust ventilation. Systems under pressure should be regularly checked for leakages. Consider the use of a work permit system e.g. for maintenance activities. Ensure exposure is below occupational exposure limits (where available).

Environmental exposure controls : Refer to local regulations for restriction of emissions to the atmosphere. See section 13 for specific methods for waste gas treatment.

8.3. Individual protection measures/Personal protective equipment

Hand protection:

Wear working gloves when handling gas containers. 29 CFR 1910.138: Hand protection

Eye protection:

Wear safety glasses with side shields. 29 CFR 1910.133: Eye and Face Protection

Skin and body protection:

Wear suitable protective clothing, e.g. lab coats, coveralls or flame resistant clothing.

Respiratory protection:

None necessary during normal and routine operations. See Sections 5 & 6.

Thermal hazard protection:

None necessary during normal and routine operations.

Other information:

Wear safety shoes while handling containers. 29 CFR 1910.136: Foot Protection.

SECTION 9: Physical and chemical properties

9.1. Information on basic physical and chemical properties

Physical state	:	Gas
Appearance	:	Clear, colorless gas.
Color	:	Colorless
Odor	:	Coal gas Odorless
Odor threshold	:	No data available
pH	:	No data available
Melting point	:	No data available
Freezing point	:	No data available
Boiling point	:	No data available
Flash point	:	No data available
Relative evaporation rate (butyl acetate=1)	:	No data available
Flammability (solid, gas)	:	No data available
Vapor pressure	:	No data available
Relative vapor density at 20 °C	:	No data available
Relative density	:	No data available
Relative gas density	:	Lighter or similar to air
Solubility	:	Water: No data available
Log Pow	:	Not applicable for gas-mixtures. Not applicable for gas-mixtures.
Auto-ignition temperature	:	No data available
Decomposition temperature	:	No data available

Viscosity, kinematic	:	No data available
Viscosity, dynamic	:	No data available
Explosion limits	:	No data available
Explosive properties	:	Not applicable (non-flammable gas).
Oxidizing properties	:	Supports combustion. Not combustible but enhances combustion of other substances.

9.2. Other information

No additional information available

SECTION 10: Stability and reactivity

10.1. Reactivity

None known.

10.2. Chemical stability

Stable under normal conditions.

10.3. Possibility of hazardous reactions

Can form explosive mixtures with flammable materials.

10.4. Conditions to avoid

None under recommended storage and handling conditions (see section 7).

10.5. Incompatible materials

Flammable materials.

10.6. Hazardous decomposition products

Under normal conditions of storage and use, hazardous decomposition products should not be produced.

SECTION 11: Toxicological information

11.1. Information on toxicological effects

Acute toxicity : Not classified

Isobutylene (115-11-7)	
LC50 inhalation rat (mg/l)	620 mg/l/4h
LC50 inhalation rat (ppm)	239620.46 ppm/4h
ATE US (gases)	239620.460 ppmV/4h
ATE US (vapors)	620.000 mg/l/4h
ATE US (dust, mist)	620.000 mg/l/4h

Oxygen (7782-44-7)	
LC50 inhalation rat (ppm)	800000 ppm/4h
ATE US (gases)	800000.000 ppmV/4h

Nitrogen (7727-37-9)	
LC50 inhalation rat (ppm)	820000 ppm/4h
ATE US (gases)	820000.000 ppmV/4h

Skin corrosion/irritation	:	Not classified
Serious eye damage/irritation	:	Not classified
Respiratory or skin sensitization	:	Not classified
Germ cell mutagenicity	:	Not classified
Carcinogenicity	:	Not classified

Isobutylene (115-11-7)	
National Toxicology Program (NTP) Status	1 - Evidence of Carcinogenicity

Reproductive toxicity	:	Not classified
Specific target organ toxicity – single exposure	:	Not classified
Specific target organ toxicity – repeated exposure	:	Not classified

Aspiration hazard	:	Not classified
Symptoms/effects after inhalation	:	Adverse effects not expected from this product.
Symptoms/effects after skin contact	:	Adverse effects not expected from this product.
Symptoms/effects after eye contact	:	Adverse effects not expected from this product.
Symptoms/effects after ingestion	:	Ingestion is not considered a potential route of exposure.
Symptoms/effects upon intravenous administration	:	Not known.
Chronic symptoms	:	Adverse effects not expected from this product.

SECTION 12: Ecological information

12.1. Toxicity

Ecology - general : No ecological damage caused by this product.

12.2. Persistence and degradability

Isobutylene (0.0005% - 1.34%), Oxygen (19.5 - 23.5%) in balance Nitrogen	
Persistence and degradability	No data available.

Isobutylene (115-11-7)	
Persistence and degradability	The substance is readily biodegradable. Unlikely to persist.

Oxygen (7782-44-7)	
Persistence and degradability	No ecological damage caused by this product.

Nitrogen (7727-37-9)	
Persistence and degradability	No ecological damage caused by this product.

12.3. Bioaccumulative potential

Isobutylene (0.0005% - 1.34%), Oxygen (19.5 - 23.5%) in balance Nitrogen	
Log Pow	Not applicable for gas-mixtures.
Log Kow	Not applicable for gas-mixtures.
Bioaccumulative potential	No data available.

Isobutylene (115-11-7)	
Log Pow	2.35
Bioaccumulative potential	Not expected to bioaccumulate due to the low log Kow (log Kow < 4). Refer to section 9.

Oxygen (7782-44-7)	
Log Pow	Not applicable for inorganic gases.
Bioaccumulative potential	No ecological damage caused by this product.

Nitrogen (7727-37-9)	
Log Pow	Not applicable for inorganic gases.
Bioaccumulative potential	No ecological damage caused by this product.

12.4. Mobility in soil

Isobutylene (0.0005% - 1.34%), Oxygen (19.5 - 23.5%) in balance Nitrogen	
Mobility in soil	No data available

Isobutylene (115-11-7)	
Ecology - soil	Because of its high volatility, the product is unlikely to cause ground or water pollution.

Oxygen (7782-44-7)	
Ecology - soil	No ecological damage caused by this product.

Nitrogen (7727-37-9)	
Ecology - soil	No ecological damage caused by this product.

12.5. Other adverse effects

Effect on ozone layer	:	None
Effect on global warming	:	No known effects from this product.
GWPmix comment	:	No known effects from this product.

SECTION 13: Disposal considerations

13.1. Disposal methods

Waste treatment methods : Contact supplier if guidance is required. Do not discharge into any place where its accumulation could be dangerous. Ensure that the emission levels from local regulations or operating permits are not exceeded.

Product/Packaging disposal recommendations : Refer to the CGA Pamphlet P-63 "Disposal of Gases" available at www.cganet.com for more guidance on suitable disposal methods.

SECTION 14: Transport information

Department of Transportation (DOT)

In accordance with DOT

Transport document description : UN1956 Compressed gas, n.o.s., 2.2

UN-No.(DOT) : UN1956

Proper Shipping Name (DOT) : Compressed gas, n.o.s.

Hazard labels (DOT) : 2.2 - Non-flammable gas

DOT Packaging Non Bulk (49 CFR 173.xxx) : 302;305

DOT Packaging Bulk (49 CFR 173.xxx) : 314;315

DOT Symbols : G - Identifies PSN requiring a technical name

DOT Packaging Exceptions (49 CFR 173.xxx) : 306;307

DOT Quantity Limitations Passenger aircraft/rail (49 CFR 173.27) : 75 kg

DOT Quantity Limitations Cargo aircraft only (49 CFR 175.75) : 150 kg

DOT Vessel Stowage Location : A - The material may be stowed ‘‘on deck’’ or ‘‘under deck’’ on a cargo vessel and on a passenger vessel.

Other information : No supplementary information available.

Special transport precautions : Avoid transport on vehicles where the load space is not separated from the driver's compartment. Ensure vehicle driver is aware of the potential hazards of the load and knows what to do in the event of an accident or an emergency. Before transporting product containers: - Ensure there is adequate ventilation. - Ensure that containers are firmly secured. - Ensure cylinder valve is closed and not leaking. - Ensure valve outlet cap nut or plug (where provided) is correctly fitted. - Ensure valve protection device (where provided) is correctly fitted.

Transportation of Dangerous Goods

Transport by sea

Transport document description (IMDG) : UN 1956 Compressed gas, n.o.s., 2.2

UN-No. (IMDG) : 1956

Proper Shipping Name (IMDG) : Compressed gas, n.o.s.

Class (IMDG) : 2.2 - Non-flammable, non-toxic gases

Limited quantities (IMDG) : 120 ml

Air transport

Transport document description (IATA) : UN 1956 Compressed gas, n.o.s., 2.2

UN-No. (IATA) : 1956

Proper Shipping Name (IATA) : Compressed gas, n.o.s.

Class (IATA) : 2

SECTION 15: Regulatory information

15.1. US Federal regulations

Isobutylene (115-11-7)
Listed on the United States TSCA (Toxic Substances Control Act) inventory

Oxygen (7782-44-7)
Listed on the United States TSCA (Toxic Substances Control Act) inventory

Nitrogen (7727-37-9)
Listed on the United States TSCA (Toxic Substances Control Act) inventory

15.2. International regulations

CANADA

Isobutylene (115-11-7)
Listed on the Canadian DSL (Domestic Substances List)

Oxygen (7782-44-7)
Listed on the Canadian DSL (Domestic Substances List)

Nitrogen (7727-37-9)
Listed on the Canadian DSL (Domestic Substances List)

EU-Regulations

Isobutylene (115-11-7)
Listed on the EEC inventory EINECS (European Inventory of Existing Commercial Chemical Substances)

Oxygen (7782-44-7)
Listed on the EEC inventory EINECS (European Inventory of Existing Commercial Chemical Substances)

Nitrogen (7727-37-9)
Listed on the EEC inventory EINECS (European Inventory of Existing Commercial Chemical Substances)

National regulations

Isobutylene (115-11-7)
Listed on the AICS (Australian Inventory of Chemical Substances) Listed on IECSC (Inventory of Existing Chemical Substances Produced or Imported in China) Listed on the Japanese ENCS (Existing & New Chemical Substances) inventory Listed on the Japanese ISHL (Industrial Safety and Health Law) Listed on the Korean ECL (Existing Chemicals List) Listed on NZIoC (New Zealand Inventory of Chemicals) Listed on PICCS (Philippines Inventory of Chemicals and Chemical Substances) Listed on INSQ (Mexican National Inventory of Chemical Substances) Listed on the TCSI (Taiwan Chemical Substance Inventory)

Oxygen (7782-44-7)
Listed on the AICS (Australian Inventory of Chemical Substances) Listed on IECSC (Inventory of Existing Chemical Substances Produced or Imported in China) Listed on the Korean ECL (Existing Chemicals List) Listed on NZIoC (New Zealand Inventory of Chemicals) Listed on PICCS (Philippines Inventory of Chemicals and Chemical Substances) Listed on INSQ (Mexican National Inventory of Chemical Substances) Listed on the TCSI (Taiwan Chemical Substance Inventory)

Nitrogen (7727-37-9)
Listed on the AICS (Australian Inventory of Chemical Substances) Listed on IECSC (Inventory of Existing Chemical Substances Produced or Imported in China) Listed on the Korean ECL (Existing Chemicals List) Listed on NZIoC (New Zealand Inventory of Chemicals) Listed on PICCS (Philippines Inventory of Chemicals and Chemical Substances) Listed on INSQ (Mexican National Inventory of Chemical Substances) Listed on the TCSI (Taiwan Chemical Substance Inventory)

15.3. US State regulations

Isobutylene (115-11-7)
U.S. - Massachusetts - Right To Know List
U.S. - New Jersey - Right to Know Hazardous Substance List
U.S. - Pennsylvania - RTK (Right to Know) List

Oxygen (7782-44-7)
U.S. - Massachusetts - Right To Know List
U.S. - New Jersey - Right to Know Hazardous Substance List
U.S. - Pennsylvania - RTK (Right to Know) List

Nitrogen (7727-37-9)
U.S. - Massachusetts - Right To Know List
U.S. - New Jersey - Right to Know Hazardous Substance List
U.S. - Pennsylvania - RTK (Right to Know) List

SECTION 16: Other information

Revision date : 03/01/2018

Other information : This Safety Data Sheet is offered pursuant to OSHA's Hazard Communication Standard, 29 CFR, 1910.1200. Other government regulations must be reviewed for applicability to this product.

Full text of H-phrases:

H270	May cause or intensify fire; oxidizer
H280	Contains gas under pressure; may explode if heated

SDS US (GHS HazCom 2012)

This Safety Data Sheet is offered pursuant to OSHA's Hazard Communication Standard, 29 CFR, 1910.1200. Other government regulations must be reviewed for applicability to this gas mixture. To the best of Calgaz's knowledge, the information contained herein is reliable and accurate as of this date; however, accruacy, suitability or completeness are not guaranteed and no warranties of any type, either express or implied, are provided. The information contained herein relates only to this specific product. If this gas mixture is combined with other materials, all component properties must be considered. Data may be changed from time to time. Be sure to consult the latest edition.

SAFETY DATA SHEET

Version 6.4
Revision Date 11/04/2019
Print Date 04/04/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Liqui-nox® phosphate-free liquid detergent

Product Number : Z273279
Brand : Aldrich

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Skin irritation (Category 2), H315
Serious eye damage (Category 1), H318
Specific target organ toxicity - repeated exposure, Inhalation (Category 2), Respiratory Tract, H373
Short-term (acute) aquatic hazard (Category 3), H402
Long-term (chronic) aquatic hazard (Category 3), H412

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)
H315 Causes skin irritation.

H318	Causes serious eye damage.
H373	May cause damage to organs (Respiratory Tract) through prolonged or repeated exposure if inhaled.
H412	Harmful to aquatic life with long lasting effects.

Precautionary statement(s)

P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P273	Avoid release to the environment.
P280	Wear protective gloves/ eye protection/ face protection.
P302 + P352	IF ON SKIN: Wash with plenty of soap and water.
P305 + P351 + P338 + P310	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER/doctor.
P314	Get medical advice/ attention if you feel unwell.
P332 + P313	If skin irritation occurs: Get medical advice/ attention.
P362	Take off contaminated clothing and wash before reuse.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.2 Mixtures

Component		Classification	Concentration
Sodium xylenesulphonate			
CAS-No. EC-No.	1300-72-7 215-090-9	Skin Irrit. 2; Eye Irrit. 2A; STOT SE 3; H315, H319, H335	>= 5 - < 10 %
Alcohols, C12-14-secondary, ethoxylated			
CAS-No.	84133-50-6	Skin Irrit. 2; Eye Dam. 1; H315, H318	>= 5 - < 10 %
Coconut diethanolamide			
CAS-No. EC-No.	8051-30-7 232-483-0	Skin Irrit. 2; Eye Dam. 1; Aquatic Acute 2; Aquatic Chronic 2; H315, H318, H401, H411	>= 5 - < 10 %
tripotassium hydrogen ethylenediaminetetraacetate			
CAS-No. EC-No.	17572-97-3 241-543-5	Acute Tox. 4; STOT RE 2; H332, H373	>= 5 - < 10 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides, Nitrogen oxides (NOx), Sulphur oxides, Potassium oxides, Sodium oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Soak up with inert absorbent material and dispose of as hazardous waste. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.
Storage class (TRGS 510): 10: Combustible liquids

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters
Contains no substances with occupational exposure limit values.

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Tightly fitting safety goggles. Faceshield (8-inch minimum). Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	No data available
f)	Initial boiling point and boiling range	No data available
g)	Flash point	()No data available
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	No data available
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
No data available

10.6 Hazardous decomposition products
Other decomposition products - No data available
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Nitrogen oxides (NOx), Sulphur oxides, Potassium oxides, Sodium oxides
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available

Dermal: No data available

No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION 12: Ecological information

12.1 Toxicity
No data available

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Harmful to aquatic life.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
Not dangerous goods

IMDG
Not dangerous goods

IATA
Not dangerous goods

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

Water	CAS-No. 7732-18-5	Revision Date
Benzenesulfonic acid, mono-C10-16-alkyl derivs., sodium salts	68081-81-2	
Sodium xylenesulphonate	1300-72-7	
Alcohols, C12-14-secondary, ethoxylated	84133-50-6	
Coconut diethanolamide	8051-30-7	
tripotassium hydrogen ethylenediaminetetraacetate	17572-97-3	

Water	CAS-No. 7732-18-5	Revision Date
Benzenesulfonic acid, mono-C10-16-alkyl derivs., sodium salts	68081-81-2	
Sodium xylenesulphonate	1300-72-7	
Alcohols, C12-14-secondary, ethoxylated	84133-50-6	
Coconut diethanolamide	8051-30-7	
tripotassium hydrogen ethylenediaminetetraacetate	17572-97-3	

New Jersey Right To Know Components

Water	CAS-No. 7732-18-5	Revision Date
Benzenesulfonic acid, mono-C10-16-alkyl derivs., sodium salts	68081-81-2	
Sodium xylenesulphonate	1300-72-7	
Alcohols, C12-14-secondary, ethoxylated	84133-50-6	

Coconut diethanolamide	8051-30-7
tripotassium hydrogen ethylenediaminetetraacetate	17572-97-3

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.4 Revision Date: 11/04/2019 Print Date: 04/04/2020

Methanol (230, 232, 233)

000000011383

Version 3.1 Revision Date 03/26/2015 Print Date 03/08/2016

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	Methanol (230, 232, 233)
MSDS Number	:	000000011383
Product Use Description	:	Solvent
Manufacturer or supplier's details	:	Honeywell International Inc. 115 Tabor Road Morris Plains, NJ 07950-2546
For more information call	:	1-800-368-0050 +1-231-726-3171 (Monday-Friday, 9:00am-5:00pm)
In case of emergency call	:	**Medical: 1-800-498-5701 or +1-303-389-1414**
	:	**Transportation (CHEMTREC): 1-800-424-9300 or +1-703-527-3887**
	:	
	:	(24 hours/day, 7 days/week)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Form	:	liquid, clear
Color	:	colourless
Odor	:	slight alcohol-like

Classification of the substance or mixture

Classification of the substance or mixture	:	Flammable liquids, Category 2 Eye irritation, Category 2A Reproductive toxicity, Category 2 Specific target organ toxicity - single exposure, Category 1, Eyes, Nervous system, Systemic toxicity

GHS Label elements, including precautionary statements

Symbol(s) :

Signal word : Danger

Hazard statements : Highly flammable liquid and vapour.
Causes serious eye irritation.
Suspected of damaging fertility or the unborn child.
Causes damage to organs.

Precautionary statements : **Prevention:**
Obtain special instructions before use.
Do not handle until all safety precautions have been read and understood.
Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
Keep container tightly closed.
Ground/bond container and receiving equipment.
Use explosion-proof electrical/ ventilating/ lighting/ equipment.
Use only non-sparking tools.
Take precautionary measures against static discharge.
Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
Wash skin thoroughly after handling.
Do not eat, drink or smoke when using this product.
Wear protective gloves/ eye protection/ face protection.

Response:
IF ON SKIN (or hair): Remove/ Take off immediately all contaminated clothing. Rinse skin with water/ shower.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
IF exposed: Call a POISON CENTER or doctor/ physician.
If eye irritation persists: Get medical advice/ attention.
In case of fire: Use dry sand, dry chemical or alcohol-resistant foam for extinction.

Storage:
Store in a well-ventilated place. Keep cool.

Store locked up.

Disposal:
Dispose of contents/ container to an approved waste disposal plant.

Carcinogenicity

No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP, IARC, or OSHA.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Formula : CH4O

Chemical nature : Substance

Chemical Name	CAS-No.	Concentration
Methanol	67-56-1	100.00 %

SECTION 4. FIRST AID MEASURES

Inhalation : Call a physician immediately. Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Use oxygen as required, provided a qualified operator is present.

Skin contact : Wash off immediately with plenty of water for at least 15 minutes. Take off contaminated clothing and shoes immediately. Wash contaminated clothing before re-use. Call a physician.

Eye contact : Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. Call a physician.

Ingestion : Call a physician immediately. Do NOT induce vomiting. Immediate medical attention is required. Never give anything by mouth to an unconscious person.

Notes to physician

Treatment : Treat symptomatically.

SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media : Alcohol-resistant foam
Carbon dioxide (CO2)
Dry chemical
Cool closed containers exposed to fire with water spray.

Unsuitable extinguishing media : Do not use a solid water stream as it may scatter and spread fire.

Specific hazards during firefighting : Flammable.
Vapours may form explosive mixtures with air.
Vapours are heavier than air and may spread along floors.
Vapors may travel to areas away from work site before igniting/flashing back to vapor source.
In case of fire hazardous decomposition products may be produced such as:
Carbon monoxide
Carbon dioxide (CO2)
Formaldehyde

Special protective equipment for firefighters : Wear self-contained breathing apparatus and protective suit.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions : Wear personal protective equipment.
Immediately evacuate personnel to safe areas.
Keep people away from and upwind of spill/leak.
Ensure adequate ventilation.
Remove all sources of ignition.
Do not swallow.
Do not breathe vapours or spray mist.
Avoid contact with skin, eyes and clothing.

Environmental precautions : Prevent further leakage or spillage if safe to do so.

Prevent product from entering drains.
Discharge into the environment must be avoided.
Do not flush into surface water or sanitary sewer system.
Do not allow run-off from fire fighting to enter drains or water courses.

Methods for cleaning up : Ventilate the area.
No sparking tools should be used.
Use explosion-proof equipment.
Contain spillage, soak up with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and transfer to a container for disposal according to local / national regulations (see section 13).

SECTION 7. HANDLING AND STORAGE

Handling

Handling : Wear personal protective equipment.
Use only in well-ventilated areas.
Keep container tightly closed.
Do not smoke.
Do not swallow.
Do not breathe vapours or spray mist.
Avoid contact with skin, eyes and clothing.

Advice on protection against fire and explosion : Keep away from fire, sparks and heated surfaces.
Take precautionary measures against static discharges.
Ensure all equipment is electrically grounded before beginning transfer operations.
Use explosion-proof equipment.
Keep product and empty container away from heat and sources of ignition.
No sparking tools should be used.
No smoking.

Storage

Requirements for storage areas and containers : Store in area designed for storage of flammable liquids.
Protect from physical damage.
Keep containers tightly closed in a dry, cool and well-ventilated place.
Containers which are opened must be carefully resealed and

kept upright to prevent leakage.
Keep away from heat and sources of ignition.
Keep away from direct sunlight.
Store away from incompatible substances.
Container hazardous when empty.
Do not pressurize, cut, weld, braze, solder, drill, grind or expose containers to heat or sources of ignition.

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Protective measures	:	Ensure that eyewash stations and safety showers are close to the workstation location.
Engineering measures	:	Use with local exhaust ventilation. Prevent vapour buildup by providing adequate ventilation during and after use.
Eye protection	:	Do not wear contact lenses. Wear as appropriate: Safety glasses with side-shields If splashes are likely to occur, wear: Goggles or face shield, giving complete protection to eyes
Hand protection	:	Solvent-resistant gloves Gloves must be inspected prior to use. Replace when worn.
Skin and body protection	:	Wear as appropriate: Solvent-resistant apron Flame retardant antistatic protective clothing. If splashes are likely to occur, wear: Protective suit
Respiratory protection	:	In case of insufficient ventilation, wear suitable respiratory equipment. For rescue and maintenance work in storage tanks use self-contained breathing apparatus. Use NIOSH approved respiratory protection.
Hygiene measures	:	When using do not eat, drink or smoke. Wash hands before breaks and immediately after handling the product. Keep working clothes separately.

Do not swallow.
Do not breathe vapours or spray mist.
Avoid contact with skin, eyes and clothing.
This material has an established AIHA ERPG exposure limit.
The current list of ERPG exposure limits can be found at http://www.aiha.org/insideaiha/GuidelineDevelopment/ERPG/Documents/2011erpgweelhandbook_table-only.pdf.

Exposure Guidelines

Components	CAS-No.	Value	Control parameters	Update	Basis
Methanol	67-56-1	TWA : time weighted average	(200 ppm)	2008	ACGIH:US. ACGIH Threshold Limit Values
Methanol	67-56-1	STEL : Short term exposure limit	(250 ppm)	2008	ACGIH:US. ACGIH Threshold Limit Values
Methanol	67-56-1	SKIN_DES : Skin designation:	Can be absorbed through the skin.	2008	ACGIH:US. ACGIH Threshold Limit Values
Methanol	67-56-1	REL : Recommended exposure limit (REL):	260 mg/m3 (200 ppm)	2005	NIOSH/GUIDE:US. NIOSH: Pocket Guide to Chemical Hazards
Methanol	67-56-1	SKIN_DES : Skin designation:	Can be absorbed through the skin.	2005	NIOSH/GUIDE:US. NIOSH: Pocket Guide to Chemical Hazards

Methanol	67-56-1	STEL : Short term exposure limit	325 mg/m3 (250 ppm)	2005	NIOSH/GUIDE:US. NIOSH: Pocket Guide to Chemical Hazards
Methanol	67-56-1	PEL : Permissible exposure limit	260 mg/m3 (200 ppm)	02 2006	OSHA_TRANS:US. OSHA Table Z-1 Limits for Air Contaminants (29 CFR 1910.1000)
Methanol	67-56-1	TWA : time weighted average	260 mg/m3 (200 ppm)	1989	Z1A:US. OSHA Table Z-1-A (29 CFR 1910.1000)
Methanol	67-56-1	STEL : Short term exposure limit	325 mg/m3 (250 ppm)	1989	Z1A:US. OSHA Table Z-1-A (29 CFR 1910.1000)
Methanol	67-56-1	SKIN_FINAL : Skin designation (Final Rule Limit applies):	Can be absorbed through the skin.	1989	Z1A:US. OSHA Table Z-1-A (29 CFR 1910.1000)

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Physical state	:	liquid, clear
Color	:	colourless
Odor	:	slight alcohol-like
pH	:	Note: Not applicable

Melting point/freezing point	:	Note: Not applicable
Boiling point/boiling range	:	64.7 °C
Flash point	:	52 °F (11 °C) Method: closed cup
Evaporation rate	:	ca. 5 Method: Compared to Butyl acetate.
Lower explosion limit	:	6 %(V)
Upper explosion limit	:	36 %(V)
Vapor pressure	:	129.32 hPa at 20 °C(68 °F)
Vapor density	:	1.11 Note: (Air = 1.0)
Density	:	0.792 g/cm3 at 20 °C
Water solubility	:	Note: completely soluble
Ignition temperature	:	464 °C
Molecular weight	:	32.04 g/mol

SECTION 10. STABILITY AND REACTIVITY

Chemical stability	:	Stable under recommended storage conditions.
Possibility of hazardous reactions	:	Hazardous polymerisation does not occur.
Conditions to avoid	:	Heat, flames and sparks. Keep away from direct sunlight.
Incompatible materials to avoid	:	Strong oxidizing agents Aluminium Magnesium May attack many plastics, rubbers and coatings.
Hazardous decomposition products	:	In case of fire hazardous decomposition products may be produced such as: Carbon monoxide Carbon dioxide (CO2) Formaldehyde

SECTION 11. TOXICOLOGICAL INFORMATION

Acute oral toxicity	:	LD50: 5,628 mg/kg Species: Rat
Acute inhalation toxicity	:	LC50: 64000 ppm Exposure time: 4 h Species: Rat
Acute dermal toxicity	:	LD50: 15,800 mg/kg Species: Rabbit
Skin irritation	:	Species: Rabbit Classification: irritating Exposure time: 24 h

Eye irritation	:	Species: rabbit eye Classification: irritating
Repeated dose toxicity	:	Species: Rat Application Route: Inhalation Test substance: Methanol Note: Developmental Toxicity NOAEL (maternal toxicity) 10,000 ppm NOAEL (developmental toxicity) 5,000 ppm Skeletal and visceral malformations.
Genotoxicity in vitro	:	Note: In vitro tests did not show mutagenic effects
Genotoxicity in vivo	:	Note: In vivo tests did not show mutagenic effects

SECTION 12. ECOLOGICAL INFORMATION

Ecotoxicity effects

Toxicity to fish	:	LC50: 29,400 mg/l Exposure time: 96 h Species: Fathead minnow
Toxicity to daphnia and other aquatic invertebrates	:	LC50: 10,000 mg/l Exposure time: 24 h Species: Daphnia (water flea)
Toxicity to bacteria	:	EC50: 43,000 mg/l Exposure time: 5 min Species: Photobacterium phosphoreum
	:	EC50: 40,000 mg/l Exposure time: 15 min Species: Photobacterium phosphoreum
	:	EC50: 39,000 mg/l Exposure time: 25 min Species: Photobacterium phosphoreum

Further information on ecology

Additional ecological information	:	Accumulation in aquatic organisms is unlikely. The product is readily degradable in the environment.

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal methods	:	Observe all Federal, State, and Local Environmental regulations.

SECTION 14. TRANSPORT INFORMATION

DOT	UN/ID No.	:	UN 1230
	Proper shipping name	:	METHANOL
	Class		3
	Packing group		II
	Hazard Labels		3

IATA	UN/ID No.	:	UN 1230
	Description of the goods	:	METHANOL
	Class	:	3
	Packaging group	:	II
	Hazard Labels	:	3 (6.1)
	Packing instruction (cargo aircraft)	:	364
	Packing instruction (passenger aircraft)	:	352
	Packing instruction (passenger aircraft)	:	Y341

IMDG	UN/ID No.	:	UN 1230
	Description of the goods	:	METHANOL
	Class	:	3
	Packaging group	:	II
	Hazard Labels	:	3 (6.1)
	EmS Number	:	F-E, S-D
	Marine pollutant	:	no

SECTION 15. REGULATORY INFORMATION

Inventories

US. Toxic Substances Control Act	:	On TSCA Inventory
Australia. Industrial Chemical (Notification and Assessment) Act	:	On the inventory, or in compliance with the inventory
Canada. Canadian Environmental Protection Act (CEPA). Domestic Substances List (DSL)	:	All components of this product are on the Canadian DSL.
Japan. Kashin-Hou Law List	:	On the inventory, or in compliance with the inventory
Korea. Toxic Chemical Control Law (TCCL) List	:	On the inventory, or in compliance with the inventory
Philippines. The Toxic Substances and Hazardous and Nuclear Waste Control Act	:	On the inventory, or in compliance with the inventory
China. Inventory of Existing Chemical Substances	:	On the inventory, or in compliance with the inventory
New Zealand. Inventory of Chemicals (NZIoC), as published by ERMA New Zealand	:	On the inventory, or in compliance with the inventory

National regulatory information

US. EPA CERCLA Hazardous Substances (40 CFR 302)	:	The following component(s) of this product is/are subject to release reporting under 40 CFR 302 when release exceeds the Reportable Quantity (RQ): Reportable quantity: 5000 lbs

	:	Methanol	67-56-1
SARA 302 Components	:	No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.	
SARA 313 Components	:	The following components are subject to reporting levels established by SARA Title III, Section 313:	
	:	Methanol	67-56-1
SARA 311/312 Hazards	:	Fire Hazard Acute Health Hazard Chronic Health Hazard	
CERCLA Reportable Quantity	:	5000 lbs	
California Prop. 65	:	WARNING: This product contains a chemical known to the State of California to cause birth defects or other reproductive harm.	
		Methanol	67-56-1
Massachusetts RTK	:	Methanol	67-56-1
New Jersey RTK	:	Methanol	67-56-1
Pennsylvania RTK	:	Methanol	67-56-1
WHMIS Classification	:	B2: Flammable liquid D1B: Toxic Material Causing Immediate and Serious Toxic Effects D2A: Very Toxic Material Causing Other Toxic Effects D2B: Toxic Material Causing Other Toxic Effects This product has been classified according to the hazard criteria of the CPR and the MSDS contains all of the information required by the CPR.	

SECTION 16. OTHER INFORMATION

		HMIS III	NFPA
Health hazard	:	2*	1
Flammability	:	3	3
Physical Hazard	:	0	
Instability	:		0

* - Chronic health hazard

Hazard rating and rating systems (e.g. HMIS® III, NFPA): This information is intended solely for the use of individuals trained in the particular system.

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text. Final determination of suitability of any material is the sole responsibility of the user. This information should not constitute a guarantee for any specific product properties.

Changes since the last version are highlighted in the margin. This version replaces all previous versions.
Previous Issue Date: 03/19/2014
Prepared by Honeywell Performance Materials and Technologies Product Stewardship Group

SAFETY DATA SHEET

1. Identification

Product identifier: NITRIC ACID

Other means of identification

Synonyms: Aqua Fortis, Azotic Acid

Product No.: 9604, V471, V231, V230, V077, 6623, 2712, 2707, 2706, 2704, H988, 5876, 5856, 5801, 5796, 1409, 9761, 9670, 9618, 9617, 9616, 9615, 9612, 9607, 9606, 9601, 9598, 9597, 5371, 20758, 20754, 20752, 20750

Recommended use and restriction on use

Recommended use: Not available.

Restrictions on use: Not known.

Manufacturer/Importer/Supplier/Distributor information

Manufacturer

Company Name:	Avantor Performance Materials, Inc.
Address:	3477 Corporate Parkway, Suite 200 Center Valley, PA 18034
Telephone:	Customer Service: 855-282-6867
Fax:	
Contact Person:	Environmental Health & Safety
e-mail:	info@avantormaterials.com

Emergency telephone number:

24 Hour Emergency: 908-859-2151

Chemtrec: 800-424-9300

2. Hazard(s) identification

Hazard classification

Physical hazards

Oxidizing liquids	Category 3
Corrosive to metals	Category 1

Health hazards

Skin corrosion/irritation	Category 1A

Unknown toxicity

Acute toxicity, oral	65 %
Acute toxicity, dermal	65 %
Acute toxicity, inhalation, vapor	100 %
Acute toxicity, inhalation, dust or mist	100 %

Unknown toxicity

Acute hazards to the aquatic environment	65 %
Chronic hazards to the aquatic environment	65 %

Label elements

Hazard symbol:

Signal word: Danger

Hazard statement: May intensify fire; oxidizer.
May be corrosive to metals.
Causes severe skin burns and eye damage.

Precautionary statement

Prevention: Wear protective gloves/protective clothing/eye protection/face protection. Wash hands thoroughly after handling. Keep only in original container. Keep away from heat. Keep/Store away from clothing/combustible materials. Take any precaution to avoid mixing with combustibles. Use only outdoors or in a well-ventilated area.

Response: In case of fire: Use water spray, foam, dry powder or carbon dioxide for extinction. Immediately call a POISON CENTER/doctor. IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower. Wash contaminated clothing before reuse. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. IF INHALED: Remove person to fresh air and keep comfortable for breathing. Absorb spillage to prevent material damage.

Storage: Store locked up. Store in corrosive resistant container with a resistant inner liner. Store in a well-ventilated place. Keep container tightly closed.

Disposal: Dispose of contents/container to an appropriate treatment and disposal facility in accordance with applicable laws and regulations, and product characteristics at time of disposal.

Other hazards which do not result in GHS classification: None.

3. Composition/information on ingredients

Mixtures

Chemical identity	Common name and synonyms	CAS number	Content in percent (%)*
NITRIC ACID		7697-37-2	65 - 70%

* All concentrations are percent by weight unless ingredient is a gas. Gas concentrations are in percent by volume.

4. First-aid measures

General information: Get medical advice/attention if you feel unwell. Show this safety data sheet to the doctor in attendance.

Ingestion: Call a physician or poison control center immediately. Do NOT induce vomiting. If vomiting occurs, keep head low so that stomach content doesn't get into the lungs.

Inhalation:	Move to fresh air. Call a physician or poison control center immediately. If breathing stops, provide artificial respiration. If breathing is difficult, give oxygen.
Skin contact:	Immediately flush with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician or poison control center immediately. Wash contaminated clothing before reuse. Destroy or thoroughly clean contaminated shoes.
Eye contact:	Immediately flush with plenty of water for at least 15 minutes. If easy to do, remove contact lenses. Call a physician or poison control center immediately. In case of irritation from airborne exposure, move to fresh air. Get medical attention immediately.

Most important symptoms/effects, acute and delayed

Symptoms:	Corrosive to skin and eyes. Causes digestive tract burns. Spray mists may cause respiratory tract irritation.

Indication of immediate medical attention and special treatment needed

Treatment:	Treat symptomatically. Symptoms may be delayed.

5. Fire-fighting measures

General fire hazards:	Strong oxidizer - contact with other material may cause fire.

Suitable (and unsuitable) extinguishing media

Suitable extinguishing media:	Water spray, fog, CO2, dry chemical, or regular foam.
Unsuitable extinguishing media:	None known.
Specific hazards arising from the chemical:	Oxidizing Contact with combustible material may cause fire. Fire may produce irritating, corrosive and/or toxic gases.

Special protective equipment and precautions for firefighters

Special fire fighting procedures:	Move containers from fire area if you can do so without risk. Use water spray to keep fire-exposed containers cool. Cool containers exposed to flames with water until well after the fire is out.
Special protective equipment for fire-fighters:	Firefighters must use standard protective equipment including flame retardant coat, helmet with face shield, gloves, rubber boots, and in enclosed spaces, SCBA. Self-contained breathing apparatus and full protective clothing must be worn in case of fire.

6. Accidental release measures

Personal precautions, protective equipment and emergency procedures:	Keep unauthorized personnel away. ELIMINATE all ignition sources (no smoking, flares, sparks or flames in immediate area). Use personal protective equipment. See Section 8 of the MSDS for Personal Protective Equipment. Ventilate closed spaces before entering them. Do not touch damaged containers or spilled material unless wearing appropriate protective clothing.

Methods and material for containment and cleaning up: Keep combustibles (wood, paper, oil, etc.) away from spilled material. Stop leak if possible without any risk. Do not absorb in sawdust or other combustible materials. Absorb spill with vermiculite or other inert material. Collect in a non-combustible container for prompt disposal. Clean surface thoroughly to remove residual contamination. Dike far ahead of larger spill for later recovery and disposal.

Notification Procedures: Dike for later disposal. Prevent entry into waterways, sewer, basements or confined areas. Stop the flow of material, if this is without risk. Inform authorities if large amounts are involved.

Environmental precautions: Do not contaminate water sources or sewer. Prevent further leakage or spillage if safe to do so. Avoid discharge into drains, water courses or onto the ground.

7. Handling and storage

Precautions for safe handling: Keep away from combustible material. Do not get in eyes, on skin, on clothing. Wash hands thoroughly after handling. Do not eat, drink or smoke when using the product. Do not taste or swallow. Never add water to acid! Never pour water into acid/base. Dilute by slowly pouring the product into water while stirring.

Conditions for safe storage, including any incompatibilities: Do not store in metal containers. Store away from heat and light. Keep away from combustible material. Keep containers closed when not in use. Store in a cool, dry place. Keep container in a well-ventilated place.

8. Exposure controls/personal protection

Control parameters

Occupational exposure limits

Chemical identity	Type	Exposure Limit values		Source
NITRIC ACID	TWA	2 ppm		US. ACGIH Threshold Limit Values (2011)
	STEL	4 ppm		US. ACGIH Threshold Limit Values (2011)
	STEL	4 ppm	10 mg/m3	US. NIOSH: Pocket Guide to Chemical Hazards (2010)
	REL	2 ppm	5 mg/m3	US. NIOSH: Pocket Guide to Chemical Hazards (2010)
	PEL	2 ppm	5 mg/m3	US. OSHA Table Z-1 Limits for Air Contaminants (29 CFR 1910.1000) (02 2006)
	TWA	2 ppm	5 mg/m3	US. OSHA Table Z-1-A (29 CFR 1910.1000) (1989)
	STEL	4 ppm	10 mg/m3	US. OSHA Table Z-1-A (29 CFR 1910.1000) (1989)

Appropriate engineering controls No data available.

Individual protection measures, such as personal protective equipment

General information: Good general ventilation (typically 10 air changes per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation, or other engineering controls to maintain airborne levels below recommended exposure limits. If exposure limits have not been established, maintain airborne levels to an acceptable level. An eye wash and safety shower must be available in the immediate work area.

Eye/face protection: Wear safety glasses with side shields (or goggles) and a face shield.

Skin protection

Hand protection: Chemical resistant gloves

Other:	Wear suitable protective clothing.
Respiratory protection:	In case of inadequate ventilation use suitable respirator. Chemical respirator with acid gas cartridge.
Hygiene measures:	Provide eyewash station and safety shower. Always observe good personal hygiene measures, such as washing after handling the material and before eating, drinking, and/or smoking. Routinely wash work clothing to remove contaminants. Discard contaminated footwear that cannot be cleaned.

9. Physical and chemical properties

Appearance

Physical state:	Liquid
Form:	Liquid
Color:	Colorless to slightly yellow
Odor:	Pungent
Odor threshold:	No data available.
pH:	1 (0.1 molar aqueous solution)
Melting point/freezing point:	-42 °C
Initial boiling point and boiling range:	122 °C
Flash Point:	Not applicable
Evaporation rate:	No data available.
Flammability (solid, gas):	No data available.
Upper/lower limit on flammability or explosive limits	
Flammability limit - upper (%):	No data available.
Flammability limit - lower (%):	No data available.
Explosive limit - upper (%):	No data available.
Explosive limit - lower (%):	No data available.
Vapor pressure:	6.4 kPa
Vapor density:	2.5
Relative density:	1.41 (20 °C)
Solubility(ies)	
Solubility in water:	Soluble
Solubility (other):	No data available.
Partition coefficient (n-octanol/water):	No data available.
Auto-ignition temperature:	No data available.
Decomposition temperature:	No data available.
Viscosity:	No data available.

10. Stability and reactivity

Reactivity:	Reacts violently with strong alkaline substances.
Chemical stability:	Material is stable under normal conditions.
Possibility of hazardous reactions:	Hazardous polymerization does not occur. Decomposes on heating.
Conditions to avoid:	Reacts violently with strong alkaline substances. Avoid contact with strong reducing agents. Excessive heat. Contact with incompatible materials.
Incompatible materials:	Alcohols. Reducing agents. Metals. Alkalies.
Hazardous decomposition products:	Nitrogen Oxides By heating and fire, corrosive vapors/gases may be formed.

11. Toxicological information

Information on likely routes of exposure

Ingestion: May cause burns of the gastrointestinal tract if swallowed.

Inhalation: May cause damage to mucous membranes in nose, throat, lungs and bronchial system.

Skin contact: Causes severe skin burns.

Eye contact: Causes serious eye damage.

Information on toxicological effects

Acute toxicity (list all possible routes of exposure)

Oral

Product: No data available.

Dermal

Product: No data available.

Inhalation

Product: No data available.

Specified substance(s):

NITRIC ACID LC 50 (Rat, 4 h): 65 mg/l

Repeated dose toxicity

Product: No data available.

Skin corrosion/irritation

Product: Causes severe skin burns.

Serious eye damage/eye irritation

Product: Causes serious eye damage.

Respiratory or skin sensitization

Product: Not a skin nor a respiratory sensitizer.

Carcinogenicity

Product: This substance has no evidence of carcinogenic properties.

IARC Monographs on the Evaluation of Carcinogenic Risks to Humans:
No carcinogenic components identified

US. National Toxicology Program (NTP) Report on Carcinogens:
No carcinogenic components identified

US. OSHA Specifically Regulated Substances (29 CFR 1910.1001-1050):
No carcinogenic components identified

Germ cell mutagenicity

In vitro
Product: No mutagenic components identified

In vivo
Product: No mutagenic components identified

Reproductive toxicity
Product: No components toxic to reproduction

Specific target organ toxicity - single exposure
Product: None known.

Specific target organ toxicity - repeated exposure
Product: None known.

Aspiration hazard
Product: Not classified

Other effects: None known.

12. Ecological information

Ecotoxicity:

Acute hazards to the aquatic environment:

Fish
Product: No data available.

Specified substance(s):
NITRIC ACID — LC 50 (Fish, 48 h): 100 - 330 mg/l Mortality

Aquatic invertebrates
Product: No data available.

Specified substance(s):
NITRIC ACID — LC 50 (Cockle (Cerastoderma edule), 48 h): 330 - 1,000 mg/l Mortality
LC 50 (Green or European shore crab (Carcinus maenas), 48 h): 180 mg/l Mortality

Chronic hazards to the aquatic environment:

Fish
Product: No data available.

Aquatic invertebrates
Product: No data available.

Toxicity to Aquatic Plants
Product: No data available.

Persistence and degradability

Biodegradation
Product: Expected to be readily biodegradable.

BOD/COD ratio
Product: No data available.

Bioaccumulative potential
Bioconcentration factor (BCF)
Product: No data available on bioaccumulation.

Partition coefficient n-octanol / water (log Kow)
Product: No data available.

Mobility in soil: The product is water soluble and may spread in water systems.

Other adverse effects: The product may affect the acidity (pH-factor) in water with risk of harmful effects to aquatic organisms.

13. Disposal considerations

Disposal instructions: Discharge, treatment, or disposal may be subject to national, state, or local laws.

Contaminated packaging: Since emptied containers retain product residue, follow label warnings even after container is emptied.

14. Transport information

DOT

UN number:	UN 2031
UN proper shipping name:	Nitric acid
Transport hazard class(es)	
Class(es):	8, 5.1
Label(s):	8, 5.1
Packing group:	II
Marine Pollutant:	No

IMDG

UN number:	UN 2031
UN proper shipping name:	NITRIC ACID
Transport hazard class(es)	
Class(es):	8, 5.1
Label(s):	8, 5.1
EmS No.:	F-A, S-Q
Packing group:	II
Marine Pollutant:	No

IATA

UN number:	UN 2031
Proper Shipping Name:	Nitric acid
Transport hazard class(es):	
Class(es):	8, 5.1
Label(s):	8, 5.1
Marine Pollutant:	No
Packing group:	II

15. Regulatory information

US federal regulations

TSCA Section 12(b) Export Notification (40 CFR 707, Subpt. D)
US. OSHA Specifically Regulated Substances (29 CFR 1910.1001-1050)
None present or none present in regulated quantities.

CERCLA Hazardous Substance List (40 CFR 302.4):
NITRIC ACID Reportable quantity: 1000 lbs.

Superfund amendments and reauthorization act of 1986 (SARA)

Hazard categories

X	Acute (Immediate)	X	Chronic (Delayed)	X	Fire		Reactive		Pressure Generating

SARA 302 Extremely hazardous substance

Chemical identity	RQ	Threshold Planning Quantity
NITRIC ACID	1000 lbs.	1000 lbs.

SARA 304 Emergency release notification

Chemical identity	RQ
NITRIC ACID	1000 lbs.

SARA 311/312 Hazardous chemical

Chemical identity	Threshold Planning Quantity
NITRIC ACID	500lbs

SARA 313 (TRI reporting)

Chemical identity	Reporting threshold for other users	Reporting threshold for manufacturing and processing
NITRIC ACID	10000 lbs	25000 lbs.

Clean Water Act Section 311 Hazardous Substances (40 CFR 117.3)

NITRIC ACID Reportable quantity: 1000 lbs.

Clean Air Act (CAA) Section 112(r) Accidental Release Prevention (40 CFR 68.130):

NITRIC ACID Threshold quantity: 15000 lbs

US state regulations

US. California Proposition 65

No ingredient regulated by CA Prop 65 present.

US. New Jersey Worker and Community Right-to-Know Act

NITRIC ACID Listed

US. Massachusetts RTK - Substance List

NITRIC ACID Listed

US. Pennsylvania RTK - Hazardous Substances

NITRIC ACID Listed

US. Rhode Island RTK

NITRIC ACID Listed

Inventory Status:

Australia AICS:	On or in compliance with the inventory
Canada DSL Inventory List:	On or in compliance with the inventory
EINECS, ELINCS or NLP:	On or in compliance with the inventory
Japan (ENCS) List:	On or in compliance with the inventory
China Inv. Existing Chemical Substances:	Not in compliance with the inventory.
Korea Existing Chemicals Inv. (KECI):	On or in compliance with the inventory
Canada NDSL Inventory:	Not in compliance with the inventory.
Philippines PICCS:	On or in compliance with the inventory
US TSCA Inventory:	On or in compliance with the inventory
New Zealand Inventory of Chemicals:	On or in compliance with the inventory
Japan ISHL Listing:	Not in compliance with the inventory.
Japan Pharmacopoeia Listing:	Not in compliance with the inventory.

16.Other information, including date of preparation or last revision

NFPA Hazard ID

Hazard rating: 0 - Minimal; 1 - Slight; 2 - Moderate; 3 - Serious; 4 - Severe
OXY: Oxidizer

Issue date:	06-04-2014
Revision date:	No data available.
Version #:	2.0
Further information:	No data available.

Disclaimer:

THE INFORMATION PRESENTED IN THIS MATERIAL SAFETY DATA SHEET (MSDS/SDS) WAS PREPARED BY TECHNICAL PERSONNEL BASED ON DATA THAT THEY BELIEVE IN THEIR GOOD FAITH JUDGMENT IS ACCURATE. HOWEVER, THE INFORMATION PROVIDED HEREIN IS PROVIDED "AS IS," AND AVANTOR PERFORMANCE MATERIALS MAKES AND GIVES NO REPRESENTATIONS OR WARRANTIES WHATSOEVER, AND EXPRESSLY DISCLAIMS ALL WARRANTIES REGARDING SUCH INFORMATION AND THE PRODUCT TO WHICH IT RELATES, WHETHER EXPRESS, IMPLIED, OR STATUTORY, INCLUDING WITHOUT LIMITATION, WARRANTIES OF ACCURACY, COMPLETENESS, MERCHANTABILITY, NON-INFRINGEMENT, PERFORMANCE, SAFETY, SUITABILITY, STABILITY, AND FITNESS FOR A PARTICULAR PURPOSE, AND ANY WARRANTIES ARISING FROM COURSE OF DEALING, COURSE OF PERFORMANCE, OR USAGE OF TRADE. THIS MSDS/SDS IS INTENDED ONLY AS A GUIDE TO THE APPROPRIATE PRECAUTIONARY HANDLING OF THE MATERIAL BY A PROPERLY TRAINED PERSON USING THIS PRODUCT, AND IS NOT INTENDED TO BE COMPREHENSIVE AS TO THE MANNER AND CONDITIONS OF USE, HANDLING, STORAGE, OR DISPOSAL OF THE PRODUCT. INDIVIDUALS RECEIVING THIS MSDS/SDS MUST ALWAYS EXERCISE THEIR OWN INDEPENDENT JUDGMENT IN DETERMINING THE APPROPRIATENESS OF SUCH ISSUES. ACCORDINGLY, AVANTOR PERFORMANCE MATERIALS ASSUMES NO LIABILITY WHATSOEVER FOR THE USE OF OR RELIANCE UPON THIS INFORMATION. NO SUGGESTIONS FOR USE ARE INTENDED AS, AND NOTHING HEREIN SHALL BE CONSTRUED AS, A RECOMMENDATION TO INFRINGE ANY EXISTING PATENTS OR TO VIOLATE ANY FEDERAL, STATE, LOCAL, OR FOREIGN LAWS. AVANTOR PERFORMANCE MATERIALS REMINDS YOU THAT IT IS YOUR LEGAL DUTY TO MAKE ALL INFORMATION IN THIS MSDS/SDS AVAILABLE TO YOUR EMPLOYEES.

ATTACHMENT 5

EMPLOYEE EXPOSURE/INJURY INCIDENT REPORT

Employee Exposure/Injury Incident Report

(completed by the CHSM or designee)

Employee:	
Office or field location:	
Incident:	
Potential or known exposure (describe):	
Physical injury or illness (describe):	

Location (city and state):		Project and Contract No.	
Date of incident:		Time of incident:	
Date incident reported:		Person to whom incident was reported:	
Weather condition during incident:		Temperature:	Precipitation:
Wind speed and direction:		Cloud cover:	

Name of materials potentially encountered (chemical exposure):

Chemical and phase (i.e., liquid, solid, gas, vapor, fume, mist), radiological, etc.:	

Describe the exposure/injury in detail and the parts of the body affected (attach extra sheets if necessary):

Describe exact location where the incident occurred:

What was the employee doing when the exposure/injury occurred? (Describe briefly as OU4 reconnaissance, soil sampling, etc.):

How did the incident occur? Describe fully the factors that led to or contributed to the incident:					
Was medical treatment given? ☐ Yes ☐ No If yes, when?					
By whom?		Name of paramedic:			
		Name of physician:			
		Other:			
Where?	On OU4		Off OU4		
If off OU4, name of hospital or clinic:					
Length of inpatient stay (dates):					
Was Integral management notified? ☐ Yes ☐ No If yes, when?					
Name and title of manager(s) notified:					
Did the exposure/injury result in permanent disability or death? ☐ Yes ☐ No					
If yes, explain:					
Number of days away from work		Number of days of restricted work activity:			
Has the employee returned to work? (Yes / No) If yes, date:					
Names of other persons affected during the incident:					
Names of persons who witnessed the incident:					
Name and title of field team leader or immediate supervisor at OU4:					
Was the operation being conducted under an established safety plan? ☐ Yes ☐ No					

If yes, attach a copy. If no, explain:	

Was personal protective equipment (PPE) used by the employee? ☐ Yes ☐ No

If yes, list items:	

Did any limitations in safety equipment or PPE affect or contribute to exposure? ☐ Yes ☐ No

If yes, explain:	

Attachments to this report:		Medical report(s) (if not confidential)		Health and safety plan
		Other relevant information		

Employee's signature		Date
OU4 safety officer's signature		Date
Project manager's signature		Date

Corporate health and safety manager review and comments

Corrective action/procedure changes carried out on the project:
Corrective actions to be taken to prevent similar incidents at other locations:

Corporate Health and Safety Manager's signature		Date

ATTACHMENT 6

NEAR-MISS INCIDENT REPORT

Near-Miss Incident Report

(completed by field staff)

Employee:	
Office or OU4 location:	

Near-Miss Incident (check one or more): Exposure ☐ Physical injury ☐ Property damage ☐

Location (city and state):		Project and Contract No.	
Date of incident:		Time of incident:	

Fully describe the incident, including how it happened, persons involved, if chemicals were involved in the incident, etc.:

Was the operation being conducted under an established safety plan? ☐ Yes ☐ No

If yes, attach a copy. If no, explain:	

Employee's signature		Date
Project Manager's signature		Date
OU4 safety officer's signature		Date

Corporate health and safety manager review and comments

Corrective action/procedure changes carried out at the OU4:		
Corrective actions to be taken to prevent similar incidents at other locations:		
Corporate Health and Safety Manager's signature		Date

ATTACHMENT 7

COVID-19 SITE AND PREVENTATIVE MEASURES PLANS

COVID-19 Field Program Management Plan

COVID-19 Site and Preventive Measure Plans

Integral Engineering, P.C. (Integral) and its subcontractors will take proper precautions to minimize to every extent possible the transmission of the SARS-CoV-2 virus (COVID-19) during site investigation activities. These activities may include site visits, construction oversight, sediment and soil sampling, groundwater monitoring, and the deployment and retrieval of in-water remote sensing instrumentation. This Field Program Management Plan may be used as an addendum to the existing project-specific Health and Safety Plan and shall remain in effect until superseded by further updates.

Guidelines presented herein are consistent with the preventive recommendations provided by the Centers for Disease Control and Prevention (CDC) (https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/), the COVID-19 planning guidance provided by the Occupational Safety and Health Administration (http://www.osha.gov/Publications/OSHA3990.pdf), and the New York State Department of Health Interim Guidance for Construction Activities during the COVID-19 Public Health Emergency (https://www.governor.ny.gov/sites/governor.ny.gov/files/atoms/files/ConstructionMasterGuidance.pdf).

Each field effort will require discussions between the project manager and client to address specific requirements associated with local orders and directives that could impact travel and health and safety. The following lists general CDC recommendations followed by steps Integral and its subcontractors will take to reduce the transmission of COVID-19.

As a precautionary measure to avoid delays, a stockpile containing 2 weeks' worth of necessary personal protective equipment (PPE) will be procured prior to mobilization and maintained onsite.

Traveling to Site

Staff on business travel will not be permitted to use public transportation until otherwise notified. Business travel by air is not recommended unless absolutely necessary. Contact Bill Locke (Integral President) or Laura Jones (Integral Vice President) for work-related air travel authorization. In most instances, staff will require rental car use for offsite commuting. In this case, Integral recommends physical distancing during travel (i.e., more than one person in a typical passenger car is not allowed).

Staff requiring rental cars for any sort of business travel, including fieldwork, are to take the following precautions when taking possession of the vehicle for the first time, and at the start of each day while renting the vehicle:

- Use a disinfecting wipe to wipe down main contact areas, including:
  - Door handles (inside and outside)
  - Steering wheel
  - Dashboard
  - Clock and entertainment surface, including knobs
  - Gear shifting knob
  - Blinker and windshield wiper knob
  - Window control switch or lever
  - Rear view mirror and mirror control knobs
  - Center console
  - Odometer acrylic screen
  - Glove compartment external door.
- Refrain from wearing the same unwashed clothes the following day or subsequent days after using a rental vehicle.
- Sanitize hands immediately after refueling and after returning the rental vehicle.

Before Entering Site

Field staff will be required to undergo body temperature screening prior to entering the site. If an individual chooses not to participate, the individual should discuss the decision with the project manager, field lead, or site safety officer. Client has the right to deny access to the facility if a temperature scan is refused.

Other actions to be taken before entering a site include the following:

- Learn the travel history of all employees and visitors to understand potential exposure. If an individual has traveled internationally or has had exposure to infected individuals within the U.S., the individual will need to self-quarantine for a minimum of 14 days. A positive COVID-19 test will also prevent staff from entering the field.
- If a staff member is feeling well but has a sick family member at home, the staff member should notify the project manager and follow CDC-recommended care (https://www.cdc.gov/coronavirus/2019-ncov/if-you-are-sick/).

- If a staff member shows any signs of a respiratory ailment (cough, sore throat, fever), he or she is required to stay home and not report to work. Symptoms of COVID-19 include fever (>100.0 °F), cough, and shortness of breath as described on the CDC website (https://www.cdc.gov/coronavirus/2019-ncov/symptoms-testing/symptoms.html). It is recommended that the individual contact a health care provider for medical advice. If COVID-19 is suspected or confirmed, staff must stay home for a minimum of 14 days.

Minimizing Chance of Exposure on the Site

- Information needed to minimize exposure and prevent the spread of COVID-19 will be included in each day's health and safety meeting. Field crew meetings should be conducted outside, if possible.
- Workers will follow site-specific Health and Safety Plan requirements for the use of PPE. PPE is not to be shared.
- If symptoms consistent with COVID-19 are noticeable during the sampling day, the employee or subcontractor should excuse him- or herself from further work, leave the site immediately, and follow CDC guidance (https://www.cdc.gov/coronavirus/2019-ncov/if-you-are-sick/).
- Workers will wash hands often with soap and water for at least 20 seconds. If soap and water are not available, hand sanitizer with at least 60 percent alcohol will be made available in multiple locations, as needed. Frequent hand-washing is recommended throughout the day (https://www.cdc.gov/handwashing/).
- Workers are to avoid touching eyes, nose, and mouth with unwashed hands.
- Workers who cough or sneeze should cover mouth and nose with a tissue or use the inside of one's elbow.
- Frequently touched objects and surfaces, such as workstations, keyboards, telephones, handrails, and doorknobs, will be cleaned and disinfected. The frequency and scope of the cleaning program for project facilities (office trailers, bathrooms, other buildings, and work areas) will be reviewed and increased as necessary. Cleaning products used will be those recommended by EPA and deemed as effective against the SARS-CoV-2 virus (https://www.epa.gov/pesticide-registration/list-n-disinfectants-use-against-sars-cov-2).
- Workers will avoid using other employees' phones or other work tools and equipment, when possible. If necessary, workers will clean and disinfect them before and after use.

Managing Visitor Access and Movement in Sampling Area

- Only staff directly involved in sample collection or equipment deployment will be permitted within the sampling zone.
- All visitors present outside the collection or deployment area will maintain at least a 6-ft distance from fellow visitors or sampling staff, even after operations are complete.
- Sequential work practices with appropriate physical distancing are to be considered and implemented wherever possible.
- Group meetings are to be minimized whenever possible. Meetings that are conducted are limited to <10 people.

Implementing Environmental Control

- Appropriate disinfectant wipes and cleaners and hand sanitizer will be made available at each job site.
- Sampling staff will clean the sampling zone and surrounding environment to ensure no sampling waste or other trash is left behind. After trash is bagged, staff will sanitize hands and exit the sampled property.

Wearing Face Coverings

The use of face coverings is another line of defense against the spread of COVID-19. On April 3, 2020, the CDC published guidelines for wearing cloth face coverings when physical distancing measures are difficult to maintain in public and work settings (https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/diy-cloth-face-coverings.html). Cloth face coverings may help slow the spread of the SARS-CoV-2 virus by individuals who do not have symptoms of illness but may be infected. The CDC has indicated that asymptomatic individuals are capable of transmitting the virus to others, especially when people are interacting in close physical proximity. Members of the sampling team are required to wear masks while onsite. Wearing a face covering, however, is not a substitute for staying home when ill, practicing good hygiene, and physical distancing whenever practical.

Cloth face coverings include nonsurgical, washable double-layered cloth masks, bandanas, and neck gators and disposable cloth masks. Individuals wearing and handling cloth masks should adhere to the following guidance:

- Wash or sanitize hands before donning or removing the covering.
- Only touch the face covering by the ear loops, ties, or bands.
- Do not put the outer surface against the face.

- Wash or sanitize hands after removing the face covering.
- Throw disposable face coverings in the trash.
- Wash reusable masks per manufacturer instructions.
- Store clean cloth masks in bags or face down on a clean surface.

Cloth masks should be replaced if they become wet in the field or when the field effort is completed.

Other face masks that offer greater protection against viruses may be available for specific field events on a case-by-case basis. An example of this type of field event may involve emergency sampling in areas of significant community-based transmission where physical distancing measures are difficult to maintain. Integral's Corporate Health and Safety Officer will work with the field team lead to identify the best type of face covering for use on the project.

COVID-19 Confirmed Case Response Plan

This section describes the management actions to be taken by field staff under different potential COVID-19 exposure scenarios. Prompt identification and isolation of potentially infectious individuals is critical in the protection of workers and visitors at the worksite.

Any individual who presents with symptoms of COVID-19 is to contact his or her personal healthcare provider. Decisions about COVID-19 confirmatory testing is at the discretion of state and local health departments or clinicians. As indicated on the CDC website, a negative test result does not rule out that an individual will not become sick later. A diagnosis of COVID-19 may not involve testing. Carriers of the virus may also be asymptomatic. As a result, this section does not differentiate between people who may or may not have symptoms.

Exposure Scenarios and Specific Actions

Person-to-person transmission of COVID-19 can occur via primary, secondary, and tertiary exposure pathways:

- Primary exposure—Employee tested positive for the virus.
- Secondary exposure—Employee who within the last 14 days had direct contact with someone outside of the field team who has been diagnosed with COVID-19.
- Tertiary exposure—Employee had direct contact with someone outside of the field team who has been quarantined as a result of close contact within the last 14 days with someone who has been diagnosed with or is being tested for COVID-19.

In the event there is a confirmed case of an employee becoming infected with COVID-19 (primary exposure), the field lead and site safety officer will take the following immediate actions:

- Instruct the employee, if still at the site, to enter home isolation immediately.
- Notify Integral's COVID-19 Response Team immediately.
- Notify those who may have been exposed to the virus based on close prolonged contact with the diagnosed individual, while maintaining confidentiality as required by the Americans with Disabilities Act (ADA).
- Restrict access to areas where the employee worked and mark them as off limits to all site personnel. Areas will be disinfected following CDC guidelines.
- Ask field staff who were in close contact with the individual to self-quarantine for 14 days (see management actions for Secondary Exposure). This scenario may delay the field event.

In the event of secondary exposure, the employee will be sent home immediately to enter a 14-day self-quarantine where the individual will self-monitor. Self-monitoring means the individual will take temperature readings twice daily to monitor for fever and remain alert to cough or difficulty breathing.

- The field lead or site safety officer will notify Integral's COVID-19 Response Team immediately.
- The field team will continue cleaning common touch areas with recommended disinfectants.
- If the employee is confirmed positive, this becomes a primary exposure scenario. Staff who were in close contact will be notified, and procedures for primary exposure will be followed.

In the event of tertiary exposure, communication with the field team is recommended. The individual will be asked to self-monitor.

- The field lead or site safety officer will notify Integral's COVID-19 Response Team immediately.
- The field team will continue cleaning common touch areas with recommended disinfectants.
- If the acquaintance is confirmed to be infected, this becomes a secondary exposure scenario. Steps for secondary exposure will be followed going forward.

All employees need to be vigilant regarding potential exposure and transmission of COVID-19. Curbing this outbreak is considered a team effort as much as the field event is itself.

Discontinuation of Home Isolation

For individuals with symptoms who are confirmed or suspected of having COVID-19, home isolation may be discontinued in accordance with CDC guidelines (https://www.cdc.gov/coronavirus/2019-ncov/if-you-are-sick/).

COVID-19 Field Program Management Plan Acknowledgement

Project Number: _____

Project Name: _____

My signature below certifies that I have read and understand the policies and procedures specified in this COVID-19 Field Management Plan.

Date	Name	Signature	Company

APPENDIX B

COMMUNITY AIR MONITORING PLAN

COMMUNITY AIR MONITORING PLAN

This community air monitoring plan (CAMP) has been prepared by Integral Engineering, P.C. on behalf of Corning Incorporated (USA) to detail the dust control and air monitoring procedures to be performed during characterization activities at the Study Area Operable Unit (OU) 4, located in Corning, New York. OU4 includes areas along the eastern and southern boundaries of the Study Area, consisting of the earthen berm and lands between the eastern boundary of OU1, OU2, OU3, and Post Creek and Highway 17/Interstate 86 to the east, as well as the berm and lands between the southern boundary of OU3 and the Chemung River. This CAMP is included as Appendix B, supplementing Study Area OU4 Flood Control Areas Characterization Work Plan (work plan).

As described in the work plan, subsurface characterization activities are planned at OU4. These activities may include sampling of soil, sediment, and surface water.

METHODS AND MITIGATION

Perimeter air monitoring generally will be conducted at two stations. One upwind and one downwind station will be established in the vicinity of characterization activities that have the potential to disturb and mobilize soil particulate matter and/or volatile organic compounds (VOCs). These are theoretical "stations" and may either be personnel with a mobile dust monitor and photoionization detector (PID) collecting data at a specified interval, or a semi-permanent but mobile fixture. The upwind and downwind locations will be modified as conditions warrant and placed in an area representative of air quality conditions. The perimeter air monitoring will be conducted in addition to the personal air monitoring described in the Section 5 of the Health and Safety Plan, included as Appendix A to the Work Plan.

Work will be generally conducted from Monday through Friday during business hours, 8 a.m. to 6 p.m. No visible dust will leave the work area, and the measures described below will ensure the safety of personnel and the community.

Water may be used for dust suppression where circumstances arise warranting such measures. Windy conditions, increased vehicle traffic, and subsurface characterization activities can cause increased suspension of particulate matter. Temporary stop work orders may be issued if conditions warrant.

Particulate monitoring is the measurement of fine particles that can include dust, smoke, and other particulate matter with a diameter less than or equal to 10 microns, also known as PM10. Air monitoring will be performed during activities that have the potential to

disturb the subsurface and suspend particles. To accurately measure PM10, a device such as miniRAM™, dataRAM™, sidePAK™, or equivalent will be used. The selected equipment will perform within the range of specifications outlined in the New York State Department of Environmental Conservation (NYSDEC) DER-10 *Technical Guidance for Site Investigation and Remediation* (NYSDEC 2010)[1].

CALIBRATION

Calibration of monitoring equipment will be performed on a daily basis prior to the start of intrusive work activities. Calibration data will be documented appropriately.

DOCUMENTATION

Data collection during monitoring will be used to provide personnel with real-time information about air quality and enable prompt mitigation actions to be undertaken if certain action levels are exceeded (outlined below in the "Action Levels" section). Data will be logged on appropriate field forms approximately every 30 minutes, or more frequently as conditions warrant during the monitoring program. Data will be provided to the regulatory agency either weekly or daily in the event action levels are exceeded and protective actions undertaken. Exceedances will be reported to NYSDEC and the New York State Department of Health (NYSDOH) the same day of the exceedance (or the next business day if the exceedance was recorded after hours) along with the reason for the exceedance, what was done to correct it, and whether the correction action was effective. Reporting associated with daily CAMP activities will be conducted in accordance with NYSDEC DER-10 *Technical Guidance for Site Investigation and Remediation* (NYSDEC 2010).

ACTION LEVELS

The following action levels are based on NYSDEC recommendations. Any exceedance of these action levels is an indicator that excessive PM10 or VOC migration may be taking place and will prompt immediate mitigation activities.

[1] NYSDEC. 2010. DER-10, Technical Guidance for Site Investigation and Remediation. New York State Department of Environmental Conservation, Division of Environmental Remediation. Updated May 3, 2010.

Concentration as Measured at Downwind Location	Duration	Action
100 µg/m^3 greater than background (upwind location) (PM10)	15 minutes sustained	Implement engineering control(s).
150 µg/m^3 greater than background (upwind location)(PM10)	Instantaneous	Stop work and reevaluate engineering control(s).
5 parts per million (ppm) above background (VOCs)	15-minute average	Halt activities and continue monitoring. Resume activities if level drops.
Greater than 5 but less than 25 ppm above background (VOCs)	15-minute average	Halt activities, identify vapor source, take corrective actions, and continue monitoring. Resume activities when level drops.
Greater than 25 ppm above background (VOCs)	Instantaneous	Shutdown work.

The safety officer and other personnel have the ability to stop work at any time if conditions warrant such action. The corporate health and safety manager and/or project manager may be consulted for feedback on mitigation actions as appropriate. The corporate health and safety manager and project manager will be informed of adverse conditions where mitigation is necessary in order to provide feedback and improvement to processes.

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN

CHARACTERIZATION WORK PLAN:
STUDY AREA OPERABLE UNIT 4 FLOOD CONTROL AREAS

NYSDEC Project ID 851046, Corning, New York

Quality Assurance Project Plan

Prepared for
Corning Incorporated
Corning, New York

Prepared by

1001 6th Avenue
11th Floor
New York, NY 10018

November 12, 2020

Affiliated with Integral Consulting Inc.

CONTENTS

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

ASP	Analytical Services Protocol
DQO	data quality objective
DUSR	data usability summary report
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
HASP	health and safety plan
Integral	Integral Engineering, P.C.
LC-MS/MS	liquid chromatography-tandem mass spectrometry
LIMS	Laboratory Information Management System
MDL	method detection limit
MRL	method reporting limit
NYSDEC	New York State Department of Environmental Conservation
OU	operable unit
PARCC	precision, accuracy, representativeness, completeness, and comparability
PFAS	per- and polyfluoroalkyl substances
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic
PTFE	polytetrafluoroethylene
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
RPD	relative percent difference
SOP	standard operating procedure
SVOC	semivolatile organic compound
TAL	target analyte list
TOC	total organic carbon
Work Plan	OU4 Characterization Work Plan

SECTION A: PROJECT MANAGEMENT

A1 INTRODUCTION

This quality assurance project plan (QAPP) describes quality assurance and quality control (QA/QC) procedures that will be used to ensure that the Study Area Operable Unit (OU) 4 characterization data results are defensible and usable for their intended purpose. The purpose of the QAPP is to provide confidence in the project data results through a system of quality control performance checks of field data entry, laboratory analysis and laboratory data reporting, and appropriate corrective actions to achieve compliance with established performance and data quality criteria. This QAPP is Appendix C to the Study Area Operable Unit 4 Flood Control Areas Characterization Work Plan (Work Plan). This QAPP has been prepared in accordance with U.S. Environmental Protection Agency (EPA) guidance for the preparation of QAPPs (USEPA 2002a).

A1.1 Project Scope and Goals

The goal of the characterization activities detailed in the Work Plan is to characterize OU4, which includes areas along the eastern and southern boundaries of the Study Area, consisting of the earthen berm and lands between the eastern boundary of OU1, OU2, OU3 and Post Creek and Highway 17/Interstate 86 to the east as well as the berm and lands between the southern boundary of OU3 and the Chemung River. The Work Plan includes descriptions of various characterization activities, including but not limited to soil borings, soil sampling, equipment decontamination; investigation-derived waste management; and reporting.

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

A1.2 Project Data Quality Objectives

This QAPP documents the QA/QC measures that will be followed during the implementation of the Work Plan activities. The objective of the data collection is to support the characterization activities at OU4. The overall quality objective for OU4 characterization is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality.

The QAPP provides a description of the analytical and reporting procedures that may be used by Integral Engineering, P.C. (Integral) and its subcontractors within OU4 for the following activities. Descriptions of field procedures are detailed in in the Work Plan:

- Surface soil sampling
- Shallow soil sampling
- Soil borings
- Sediment sampling
- Surface water sampling
- Laboratory analysis
- 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 they are of sufficient quality to make decisions for the project. The DQOs process is described in the subsequent subsection.

A1.2.1 Data Quality Objectives

The DQO process and quality assurance objectives for the project are presented in this section. The QA/QC procedures were developed to ensure that the analytical data collected through implementation of the Work Plan are of known and acceptable quality.

Primary DQOs will include completion of OU4 characterization activities to adequately confirm the presence or absence of constituents of concern at concentrations greater than reasonable quantitation limits, evaluate the chemical analysis results against background levels and against applicable regulatory criteria or guidance. Soil analysis results will be compared to 6 NYCRR Table 375-6.8(b), sediment analysis results will be compared to NYSDEC Screening and Assessment of Contaminated Sediment, Table 5 (NYSDEC 2014), and surface water analysis results will be compared to 6 NYCRR 703.5.

To achieve the DQOs, quality assurance measures will be implemented throughout the project to ensure that the data meet selectivity, precision, accuracy/bias, representativeness, comparability, and completeness criteria. This will be accomplished through the collection of field quality control samples, including field replicate samples, and the calibration of field and laboratory equipment.

The DQOs will be accomplished by ensuring the following analytical and quality assurance objectives are met:

- Standard methods to prepare and analyze samples are used
- Usable and defensible analytical results are obtained

- Procedures for the ongoing control and evaluation of measurement data quality are in place
- Data quality measures in terms of selectivity, precision, accuracy, completeness, representativeness, and comparability are assessed to determine whether the data meet the project objectives and can be used for their intended purpose.

The Integral quality assurance chemist will track data, from collection of samples through login at the laboratory to delivery by results report and electronic data deliverable (EDD); oversee data usability summary report (DUSR) preparation; 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 quality control practices discussed in this section. The basis for assessing selectivity, precision, accuracy, representativeness, comparability, and completeness is discussed in the laboratory quality assurance manual (Attachment 1 of this QAPP).

A2 PROJECT ORGANIZATION

This section presents the organizational structure for the field activities, including task management, oversight, field and laboratory management, data management, and health and safety. Task roles and associated responsibilities are described below.

A2.1 Key Task Personnel

- Integral Principal-in-Charge—Marcia Greenblatt is the principal-in-charge and has overall responsibility for senior technical review and oversight of the field activities, ensuring appropriate design, and implementation of the characterization to meet feasibility project objectives.
- Integral Project Manager—Jeff Marsh is the project manager. Mr. Marsh will work closely with all other team members and serve as the primary point of contact to ensure coordination between the New York State Department of Environmental Conservation (NYSDEC) and the Integral team.
- Integral Field Manager—The field manager, TBD, is responsible for overseeing the planning and coordination of the field activities and for all aspects of sample collection activities to ensure that appropriate sampling, quality assurance, and documentation procedures are used. Field team leaders will be assigned for individual tasks, as appropriate. The field manager will report to Integral's project manager.

- Integral Quality Assurance Chemist—Glenn Esler is responsible for providing overall quality assurance support for the field activities and for coordinating with the analytical lab(s) to ensure that QAPP requirements are followed. Mr. Esler is responsible for coordinating the validation of laboratory data; communicating data quality issues to the data users; and working with data users and the project manager to address any data limitations. Mr. Esler is also responsible for coordinating with the laboratory and tracking the laboratory's progress; verifying that the laboratory has implemented the requirements of the QAPP; addressing quality assurance issues related to the laboratory analyses; ensuring that laboratory capacity is sufficient to undertake the required analyses in a timely manner; and addressing scheduling issues related to laboratory analyses. Mr. Esler will report directly to Integral's project manager, and will work closely with Integral's field manager to ensure that the objectives of the QAPP are met. Resumes of Integral data validation personnel are included as Attachment 2.
- Integral Database Administrator—The database administrator, TBD, will have primary responsibility for data management and database maintenance and development. The database administrator will be responsible for overseeing and/or conducting the following activities: establishing storage formats and procedures appropriate for all data collected during the field activities; working with the field crew, laboratory, and data validator to ensure all data entries are correct and complete and are delivered in the correct format; maintaining the integrity and completeness of the database; and providing data summaries to data users in the required formats for interpretation and reporting. The database administrator will report directly to Integral's project manager and will work closely with the field manager and quality assurance chemist.
- Integral Corporate Health and Safety Manager—Matt Behum is Integral's corporate health and safety manager and will be responsible for oversight of the health and safety program that will be implemented during the field activities.
- Integral OU4 Safety Officer—The OU4 safety officer, TBD, will serve as the point of contact for safety and health concerns and will be responsible for the implementation and compliance of the health and safety plan (HASP) by all Integral staff and subcontractors.
- EurofinsTestAmerica Project Manager—John Schove, TestAmerica's project manager, will be responsible for the oversight of all laboratory functions and operations, including coordination with/between Integral and the laboratory quality manager.
- Eurofins TestAmerica Laboratory Quality Manager—The TestAmerica laboratory quality manager's, Brad Prinzi, responsibilities include the oversight of the laboratory's quality systems and ensuring that all tasks performed by the laboratory and Eurofins TestAmerica field personnel are conducted in compliance with state, federal, and industry standards, as well as the requirements of this QAPP.

A2.2 Subcontractors

If subcontractors are required, the project manager will coordinate with the subcontractors. The field team manager will direct the subcontractors in the field in accordance with their specific scope of work.

A3 TRAINING AND CERTIFICATION

Integral has assembled a project team with the requisite experience and technical skills to successfully complete the OU4 characterization. All consultant team personnel involved in sample collection have extensive environmental sampling experience. Minimum training and certification requirements for laboratory personnel are described in the laboratory quality assurance manual (Attachment 1 of this QAPP).

Information pertaining to project-specific training and certification, including medical monitoring, the Occupational Safety and Health Administration's Hazardous Waste Operations and Emergency Response standard training, first aid/cardiopulmonary resuscitation, equipment operation, and associated records and documentation, can be found in the HASP. Documentation of training will be maintained in personnel files.

SECTION B: DATA GENERATION AND ACQUISITION

B1 SAMPLING METHODS

Sampling and decontamination methods used to collect samples are described in detail in Section 5 of the Work Plan.

Per- and polyfluoroalkyl substances (PFAS) sampling and analysis procedures will conform to the guidelines provided in *Guidelines for Sampling and Analysis of PFAS under NYSDEC's Part 375 Remedial Programs* (NYSDEC 2020), and 1,4-dioxane sampling and analysis procedures will conform to the guidelines provided in *Sampling for 1,4-Dioxane and PFAS under DEC's Part 375 Remedial Programs* (NYSDEC 2019)[1].

The laboratory holds Environmental Laboratory Accreditation Program (ELAP) certification for perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) in drinking water by EPA Method 537.1 and their standard operating procedure (SOP) for PFAS analysis is included in Attachment 1. Samples will be analyzed for the compounds listed in Table C2-3 by liquid chromatography-tandem mass spectrometry (LC-MS/MS) using methodologies based on EPA Method 537.1. The method detection limits (MDLs) for the PFAS compounds are also included in Table C2-3.

Precautions regarding sampling for PFAS will include the following:

- Use of new clothing or footwear, clothing containing polytetrafluoroethylene (PTFE), or clothing treated with PFAS materials will be avoided.
- Items such as insect repellents, fluorinated hygiene products, waterproof gear, and stain-resistant materials are prohibited in the sampling area.
- Acceptable materials for sampling collection are stainless steel, polyvinyl chloride, silicone, and polypropylene.
- Stainless-steel hand augers or shovels with no coatings are permitted for soil sample collection.
- Soil samples must be mixed in a stainless-steel bowl before being placed in sample containers.
- Teflon™-free sampling equipment must be used.
- Chemical ice bags are prohibited.
- Nitrile gloves must worn at all times.

[1] The 2020 guidelines supersede the 2019 guidelines for PFAS sampling.

- Food is not allowed in the sampling area.

Soil samples will be collected with a cleaned trowel and placed into high-density polyethylene containers. Sampling equipment will be decontaminated in the standard two-step process of washing with detergent (Alconox® or Liquinox®, only) and rinsing with laboratory-certified PFAS-free water. Equipment blank samples will be collected once per day for PFAS and samples will be shipped using only regular (water) ice.

B2 SAMPLE HANDLING AND CUSTODY

The principal documents used to identify samples and document sample possession will be field logbooks and chain-of-custody records. Custody will be documented for all samples at all stages of the analytical or transfer process. Samples are in custody if they are in the view of the field team, stored in a secure place with restricted access, or placed in a container secured with custody seals. A chain-of-custody record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the chain-of-custody will be included in laboratory and QA/QC reports. Additional details regarding chain-of-custody procedures to be followed for this sampling event are provided in SOP AP-03 (Appendix D of the Work Plan).

Upon receipt of samples at the laboratory, the physical integrity of the containers and seals will be checked, and the samples will be inventoried by comparing sample labels to those on the chain-of-custody forms. The laboratory will include a copy of the chain-of-custody and shipping container receipt forms in the final data package. Any breaks in the chain-of-custody or nonconformances will be noted and reported in writing to Integral's quality assurance chemist within 24 hours of receipt of the samples, and Corning Incorporated will be notified. The laboratory quality assurance plan (Attachment 1 of this QAPP) includes procedures used for accepting custody of samples and documenting samples at the laboratory. The laboratory project manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratory. A copy of a Eurofins TestAmerica chain of custody is found in Attachment 3 of the QAPP.

All samples will be stored in accordance with Table C2-1. A subsample of each sample will be archived frozen for possible future analysis. The laboratory will maintain chain-of-custody documentation and documentation of proper storage conditions for the entire time that the samples are in its possession.

The laboratory will not dispose of the samples for any of the phases of this project until authorized to do so by Integral's quality assurance chemist. After authorization is obtained, the laboratory will dispose of samples, as appropriate, based on matrix, analytical results, and information received from the client.

B3 ANALYTICAL METHODS

Samples to be collected for the OU4 characterization include surface soil, shallow soil, subsurface non-native soil, and subsurface native soil. The specific analyses to be measured, analytical methods, and holding times are presented in Table C2-1. The following is a summary of laboratory analyses for each matrix to be sampled:

- **All surface, shallow, and boring soil samples**—Target analyte list (TAL) metals, semivolatile organic compounds (SVOCs). 1,4-dioxane and PFAS for 20 percent of samples.
- **Surface Water**—TAL metals (total and dissolved), SVOCs, total suspended solids, total dissolved solids, total organic carbon (TOC), and dissolved organic carbon. 1,4-dioxane and PFAS for 20 percent of samples.
- **Sediment**—TAL metals, SVOCs, TOC, grain size, sulfides, nitrates, carbonates, ancillary parameters to characterize bioavailability. 1,4-dioxane and PFAS for 20 percent of samples.

Eurofins TestAmerica Buffalo (Amherst, New York) will be responsible for performing the majority of analyses. If necessary, other certified laboratories in the Eurofins TestAmerica network may be used to meet analytical capacity and project objectives.

B4 QUALITY CONTROL

Processes established to ensure quality both in the field and in the laboratory are described below.

B4.1 Field Quality Control Samples

Field quality control samples will be used to assess sample variability and evaluate potential sources of contamination. The types of quality control samples that will be collected for the field activities are described in this section. If quality control problems are encountered, they will be brought to the attention of Integral's quality assurance chemist. Corrective actions, if appropriate, will be implemented to meet the project's data quality indicators.

Field quality control samples for soil, sediment, and surface water will be field duplicate samples, field blanks, equipment rinsate blanks, and temperature blanks. The frequency of collection of the field quality control samples is outlined in Table C2-2. The following quality control samples will be collected in the field and analyzed by the analytical laboratory:

- Field duplicate samples will be collected and analyzed to assess the variability associated with sample processing and laboratory variability. Blind field split samples

will be collected at a minimum frequency of 1 field split sample per 20 soil, sediment, groundwater, and surface water samples. Samples will be assigned unique numbers and will not be identified as field splits to the laboratory.

- Equipment rinsate blanks will be collected to help identify possible contamination from the sampling environment or from the sampling equipment. All equipment rinsate blank samples will be clearly noted in the field logbook (e.g., sample identifier, equipment type, date and time of collection, and analysis).
  - A minimum of one equipment blank (rinsate) will be collected for each kind of sampling equipment used for chemical analyses. A rinsate blank will be collected at every 20 locations per type of equipment used. For PFAS, an equipment blank will be collected daily, with a minimum of 1 per 20 samples. One equipment rinsate blank will be prepared for each individual analysis.
- Deionized water (field) blanks are prepared in the field to evaluate potential background concentrations present in laboratory-grade deionized water used for the equipment rinsate blank. Field blanks will be collected at a minimum frequency of one per day.
- Temperature blanks will be used by the laboratory to verify the temperature of the samples upon receipt at the testing laboratory. Temperature blanks will be prepared at the testing laboratory by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank will be included with each sample cooler shipped to the testing laboratory.

B4.2 Laboratory Quality Control

Extensive and detailed requirements for laboratory quality control procedures are provided in the EPA method protocols that will be used for this project (Table C2-1). Every method protocol includes descriptions of quality control procedures, and many incorporate additional quality control requirements by reference to separate quality control chapters in the protocols. Quality control requirements include control limits and requirements for corrective action in many cases. Quality control procedures will be completed by the laboratory, as required in each protocol and as indicated in this QAPP.

For chemical analyses, the frequency of analysis for laboratory control samples, matrix spike samples, matrix spike duplicates or laboratory duplicates, and method blanks will be 1 for every 20 samples or 1 per extraction batch, whichever is more frequent. Internal standards and/or surrogates will be added to every field sample and quality control sample, as required by the analytical methods. Calibration procedures will be completed at the frequency specified in each method description. As required for EPA SW-846 methods, performance-based control limits have been established by the laboratory (USEPA 2014). These and all other control limits

specified in the method descriptions will be used by the laboratory to establish the acceptability of the data or the need for reanalysis of the samples.

B5 DATA QUALITY INDICATORS

Data quality indicators, such as the precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters (USEPA 2002a), and analytical sensitivity will be used to assess conformance of data with quality control criteria. PARCC parameters are commonly used to assess the quality of environmental data.

B5.1 Precision

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of matrix spike duplicates, laboratory duplicates, and field duplicates. Precision is expressed in terms of the relative standard deviation for three or more measurements and the relative percent difference (RPD) for two measurements. The following equation is used to calculate the RPD between measurements:

$$\text{RPD} = \frac{|C_1 - C_2|}{\frac{(C_1 + C_2)}{2}} \times 100$$

Where:

RPD = relative percent difference
C_1 = first measurement
C_2 = second measurement

The relative standard deviation is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

B5.2 Accuracy

Accuracy or bias represents the degree to which a measured concentration conforms to the reference value. The results for matrix spikes, laboratory control samples, field blanks, and method blanks will be reviewed to evaluate bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%\text{R} = \frac{M - U}{C} \times 100$$

Where:

%R	=	percent recovery
M	=	measured concentration in the spiked sample
U	=	measured concentration in the unspiked sample
C	=	concentration of the added spike

The following calculation is used to determine percent recovery for a laboratory control sample or reference material:

$$\%R = \frac{M}{C} \times 100$$

Where:

%R	=	percent recovery
M	=	measured concentration in the reference material
C	=	established reference concentration

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analytes detected in field or method blanks will be evaluated as potential indicators of bias.

B5.3 Representativeness

Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling stations and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

B5.4 Completeness

Completeness will be calculated as the ratio of usable data (i.e., unqualified data and J-qualified data) to generated data, expressed as a percentage. Completeness will be calculated for each suite of analytes for each sample type and sampling event. The target for completeness for all components of this project is 90 to 100 percent.

B5.5 Comparability

Comparability is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be addressed through the use

of field and laboratory methods that are consistent with methods and procedures recommended by EPA, and by statistical evaluation of the data.

Additional laboratory quality control procedures will be evaluated to provide supplementary information regarding overall quality of the data, performance of instruments and measurement systems, and sample-specific matrix effects.

Quality control samples and procedures are specified in each method protocol (Table C2-1). All quality control requirements will be completed by the laboratory as described in the protocols, including the following (as applicable):

- Instrument tuning
- Initial calibration
- Initial calibration verification
- Continuing calibration
- Calibration or instrument blanks
- Method blanks
- Laboratory control samples
- Surrogates
- Internal standards
- Serial dilutions
- Matrix spikes
- Matrix spike duplicates or laboratory duplicates.

To alert the data user to possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated quality control samples or procedures do not meet control limits. Laboratory control limits for the methods that will be used for this project are provided in the laboratory's quality assurance plan (to be provided under separate cover, as requested).

MDLs are statistically derived and reflect the minimum measured concentration at which an analyte can be detected in a clean matrix with 99 percent confidence that a false positive result has not been reported (i.e., that the measured concentration is distinguishable from method blank results). Method reporting limits (MRLs) will be established at levels above the MDLs for the project analytes. These values are based on the laboratory's experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system. The

concentration of the lowest standard in the initial calibration curve for each analysis is at the level of the MRL. This allows reliable quantification of concentrations to the MRL. Test methods will be in accordance with the 2017 Clean Water Act Method Update Rule, effective September 27, 2017, which contains revised MDL definitions.

Analyte concentrations for this project will be reported to the MDL. Analytes detected at concentrations between the MRL and the MDL will be reported with a "J" qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range). Non-detects will be reported at the MRL. The MDL will be adjusted by the laboratory, as necessary, to reflect sample dilution or matrix interference. Laboratory MRLs and MDLs are found in Table C2-3.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the laboratory in accordance with the requirements identified in the laboratory SOPs and manufacturer instructions. In addition, each of the specified analytical methods provides protocols for proper instrument setup and tuning, and critical operating parameters. Instrument maintenance and repair will be documented in the maintenance log or record books.

Maintenance and calibration of the instruments to be used for field parameter measurements will be completed, as described in the manufacturers' instructions and the SOPs for their use (to be provided under separate cover, as requested).

B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY OF CALIBRATION

Laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter before beginning each analysis. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratory's quality assurance plan.

All calibration standards will be obtained from either the EPA repository or a commercial vendor, and the laboratory will maintain traceability back to the National Institute of Standards and Technology. Stock standards will be used to make intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and quality control purposes.

The quality of laboratory water used for decontamination will be documented at the laboratory. Certifiably clean and documented sample containers will be provided by the laboratory. All containers will be visually inspected prior to use by field staff, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratory are provided in the quality assurance plan (Attachment 1 of this QAPP). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (for supplies used in the field) or the laboratory.

B9 NON-DIRECT MEASUREMENTS

In order to inform and support the field sampling plan approach and methodology, a records review and preliminary review of historic aerial photos have been conducted. Existing chemical data from previous samples collected by NYSDEC were also used to design this characterization. Historical data were not reviewed for quality assurance.

B10 DATA MANAGEMENT

Data for this project will be generated in the field and at the laboratory. The final repository for all sample information will be a project database. Procedures to be used to transfer data from the point of generation to the project database are described in this section.

B10.1 Field Data

Data that are generated during sample collection will be manually entered into the field logbook and field sampling forms. Additional details regarding field documentation procedures to be followed for this sampling event are provided in SOP AP-02 (Appendix D of the Work Plan). Data from these sources will be entered into the project database directly from the field logbook and field sampling forms. These data include station location coordinates, station names, sampling dates, sample identification codes, additional station and sample

information (e.g., sample type, field duplicate number), and results. All entries will be reviewed for accuracy and completeness by a second individual, and any errors will be corrected before the data are approved for release to data users.

B10.2 Laboratory Data

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

In addition, Eurofins TestAmerica will provide an EDD that complies with NYSDEC's Electronic Warehouse Standards for all samples, with quality control sample data to be utilized during the data review/validation activities. The EDD with validated data will be provided to NYSDEC.

A variety of manually entered and electronic instrument data are generated at the laboratory. Data are manually entered into:

- Standard logbooks
- Storage temperature logs
- Balance calibration logs
- Instrument logs
- Sample preparation and analysis worksheets
- Maintenance logs
- Individual laboratory notebooks
- Tables of results for conventional analyses (i.e., total solids).

The Laboratory Information Management System (LIMS) is the central data management tool for the laboratory data. All manual data entry into the LIMS is reviewed at the laboratory. All data collected from each laboratory instrument, either manually or electronically, are reviewed and confirmed by analysts before reporting. The LIMS is used for every aspect of sample processing, including sample log-in and tracking, instrument data storage and processing, generation of data reports for sample and quality control results, and preparation of EDDs.

Laboratory data will be entered directly into the project database from the EDD. A database printout will be used to verify 10 percent of the database entries against the laboratory data packages.

SECTION C: ASSESSMENT AND OVERSIGHT

A formal chain of communication has been established for this project to optimize the flow of information and to keep the management team apprised of activities and events. The field team leaders and the chemical laboratory will stay in close verbal contact with the Integral project manager and quality assurance chemist, respectively, during all phases of the project. This level of communication will serve to keep the management team appraised of activities and events, and will allow for informal but continuous project oversight.

Assessment activities will include readiness reviews prior to sampling and prior to release of the final data to the data users, and internal review while work is in progress. An informal technical systems audit may be conducted if problems are encountered during any phase of this project.

Readiness reviews are conducted to ensure that all necessary preparations have been made for efficient and effective completion of each phase of project work. The first readiness review will be conducted prior to field sampling. The field coordinator will verify that all field equipment is ready for transfer to OU4. The field coordinator will also verify that the field team and any subcontractors have been scheduled and fully briefed on field methods and objectives, and that the contracts for the subcontractors have been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed before final data are released for use. The database administrator (or designee) will verify that all results have been received from the laboratory, data validation and data quality assessments have been completed for all of the data, and that data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the data manager (or designee) or the Integral quality assurance chemist (or designee). All data included in the data reports will have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the project manager and the data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this project will be completed throughout the course of all sampling, laboratory, data validation, data management, and data interpretation activities to ensure that every phase of work is accurate and complete and follows the quality assurance procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the Integral project manager. NYSDEC will be notified of any problems that may affect the final outcome of the project.

The laboratory has implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Each phase of work is reviewed by a supervisor before it is approved for release. Details are provided in the laboratory's quality assurance manual (Attachment 1 of this QAPP).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. If completed, these audits will be conducted by the Integral quality assurance chemist or his/her designee or by the laboratory's quality assurance manager. These audits may consist of on-location reviews of any phase of field or laboratory activities or data management. Results of any audits will be provided in the final data report.

Corrective actions will be required if deviations from the methods or quality assurance requirements established in this QAPP are encountered. When a nonconformance is identified, corrective action will be taken immediately, if possible. The project manager will be contacted and, if necessary, will provide assistance in resolving the issue. A formal corrective action plan is not required for this project. However, any nonconformance issue that ultimately affects the quality of the data or results in a change of scope in the work described in the QAPP will be documented in the field log or in a memorandum to the project manager. This documentation will serve as a corrective action report. A description of the nonconformance issue, the attempted resolution, and any effects on data quality or usability will be provided in the appropriate data report.

The laboratory has implemented a routine system of reporting nonconformance issues and their resolution. These procedures are described in the laboratory's quality assurance manual (Attachment 1 of this QAPP). Laboratory nonconformance issues will also be described in the data report if they affect the quality of the project data.

SECTION D: DATA VALIDATION AND USABILITY

D1 DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratory will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in a DUSR. The data validation reports will summarize all significant data quality issues for the sampling event and will be attached to the project characterization report.

D1.1 Data Review, Verification, and Validation

Field and laboratory data for this project will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

Data verification and validation of laboratory data will be performed by in accordance with DER-10 Appendix 2B, EPA *Guidance on Environmental Data Verification and Validation* (USEPA 2002b), EPA *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (USEPA 2009), and EPA inorganic and organic data validation methods, using USEPA Region 2 SOP modifications (USEPA 2017a,b).

Laboratory control limits will be used during data validation to assess laboratory control samples, matrix spike samples, and matrix spike or laboratory duplicates. Data may be qualified as estimated if control limits for any other quality control sample or procedure do not meet laboratory control limits.

Results for field duplicates will be evaluated using a target control limit of 50%. Data will not be qualified as estimated if the target control limit is exceeded, but RPD results will be tabulated, and any exceedances will be discussed in the data report. Equipment and field blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in the functional guidelines and Region 2 SOP modifications for data review (USEPA 2017a,b). Sample preparation blanks will be reviewed and qualified in accordance with the functional guidelines and Region 2 SOP modifications for data review (USEPA 2017a,b).

Data will be rejected if control limits for acceptance of data are not met, as described in the functional guidelines and Region 2 SOP modifications for data review (USEPA 2017a,b).

D1.2 Verification and Validation Methods

Field data will be verified during preparation of samples and chain of custody forms. Field data and chain of custody forms will be reviewed by the field coordinator on a daily basis and/or after the field effort is complete. After field data are entered into the project database, 100 percent verification of the entries will be completed to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is released for use.

The accuracy and completion of laboratory entries to the database will be verified at the laboratory when the EDDs are prepared and again as part of data validation. Ten percent of entries to the database from laboratory EDDs will be checked against laboratory data report packages. In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use.

D1.3 Reconciliation with User Requirements

The goal of data validation is to determine the quality of each data point and to identify data points that do not meet the project criteria. Nonconforming data may be qualified as estimated, or rejected as unusable, during data validation if criteria for data quality are not met. An explanation of the rejected data will be included in the DUSR. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the DUSR, as applicable.

Data qualified as estimated will be appropriately qualified in the final project database. These data may be less precise or less accurate than unqualified data. Rejected data will not be used for any purpose. The data users, in cooperation with the Integral project manager and quality assurance chemist, are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses for this characterization. The data quality discussion in the DUSR will include available information regarding the direction or magnitude of bias or the degree of imprecision for qualified data to facilitate the assessment of data usability and will include an explanation of any rejected data. The DUSR and characterization report will also include a discussion of data limitations and their effect on data interpretation activities.

D2 DATA REPORTING

The DUSR will be prepared in accordance with NYSDEC DER-10 Appendix 2B. 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.

Final and validated/reviewed analytical data, including applicable qualifiers, will be summarized in tables for associated project characterization summary reports.

SECTION E: REFERENCES

NYSDEC. 2014. Screening and Assessment of Contaminated Sediment. New York State Department of Environmental Conservation, Division of Fish, Wildlife, and Marine Resources, Bureau of Habitat. June 2014.

NYSDEC. 2019. Sampling for 1,4-Dioxane and PFAS under DEC's Part 375 Remedial Programs. New York State Department of Environmental Conservation. June 2019.

NYSDEC. 2020. Guidelines for Sampling and Analysis of PFAS under NYSDEC's Part 375 Remedial Programs. New York State Department of Environmental Conservation. January 2020.

USEPA. 2002a. Guidance for quality assurance project plans. EPA QA/G-5. EPA/240/R-02/009. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA. 2002b. Guidance on environmental data verification and validation. EPA AQ/G-8. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA. 2009. Guidance for labeling externally validated laboratory analytical data for Superfund use. USEPA-540-R-08-005. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, DC.

USEPA. 2014. SW-846 on-line, test methods for evaluating solid waste - physical/chemical methods. Available at http://www.epa.gov/epaoswer/hazwaste/test/main.htm. U.S. Environmental Protection Agency, Washington, DC.

USEPA. 2017a. National functional guidelines for inorganic superfund methods data review (using USEPA Region 2 SOP modifications). EPA-540-R-2017-001. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation, Washington, DC.

USEPA. 2017b. National functional guidelines for organic superfund methods data review (using USEPA Region 2 SOP modifications). EPA-540-R-2017-002. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation, Washington, DC.

TABLES

Table C2-1. Analytical Methods, Preservation, and Holding Times

Analysis	Analytical Methods	Minimum Volume and Container	Preservation	Holding Time[a]
Soil/Sediment				
Nitrate/Nitrite-N	EPA 353.2	50 grams, Wide-mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4±2ºC	28 days
Sulfides	SM 4500-S2 F		4±2ºC	7 days
Grain Size	ASTM D422	Full, 16 ounce glass w/Fluoropolymer Resin / Teflon®-lined lid	4±2ºC	180 days
Total organic carbon (TOC)	Lloyd Kahn	50 grams,4 ounce glass w/Fluoropolymer Resin / Teflon®-lined lid	4±2ºC	14 days
TAL Metals	SW846 6010C/7471A	10 grams, Wide-mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4±2ºC	6 months (Hg 28 days)
SVOCs	SW846 8270D	50 grams, Wide-mouth glass w/Fluoropolymer Resin / Teflon®-lined lid	4±2ºC	14/40 days[b]
PFAS	EPA 537.1M ID[e]	100 grams, Wide-mouth LDPE plastic	4±2ºC	14/28 days[c]

Table C2-1. Analytical Methods, Preservation, and Holding Times

Analysis	Analytical Methods	Minimum Volume and Container	Preservation	Holding Time[a]
Surface Water/Field Blanks				
Dissolved organic carbon (DOC)	SM 5310D	3-40 mL, glass vials w/Teflon®-lined septum	4±2ºC, HCl to pH < 2	28 days
Total dissolved solids (TDS)	SM 2540C	500 mL, Wide-mouth glass w/Teflon®-lined cap	4±2ºC	7 days
Total suspended solids (TSS)	SM 2540D	1000 mL, Wide-mouth glass w/Teflon®-lined cap	4±2ºC	7 days
Total organic carbon (TOC)	SM 5310D	3-40 mL, glass vials w/Teflon®-lined septum	4±2ºC, HCl to pH < 2	28 days
TAL Metals (total)	SW846 6010C/7470A	250 mL, Polyethlyene	4±2ºC, HNO_3 to pH < 2	6 months (Hg 28 days)
TAL Metals (dissolved)	SW846 6010C/7470A	250 mL, Polyethlyene	4±2ºC, Field filtered, HNO_3 to pH < 2	6 months (Hg 28 days)
SVOCs	SW846 8270D	1000 mL, Wide-mouth glass w/Teflon®-lined cap	≤6ºC	7/40 days[d]
PFAS	EPA 537.1M ID	2-250 mL, HPDE plastic	≤6ºC	14/28 days[e]
1,4-Dioxane	SW846 8270D SIM ID	2-1000 mL Wide-mouth glass w/Teflon®-lined cap	≤6ºC	7/40 days[d]

Notes:

ASTM = American Society of Testing and Materials
EPA = U.S. Environmental Protection Agency
HDPE = high-density polyethylene
ID = isotope dilution
PFAS = per- and polyfluoroalkyl substances
SIM = selected ion monitoring
SM = Standard Method
SVOC = semivolatile organic compound
SW846 = Test Methods for Evaluating Solid Waste: Physical/Chemical Methods
TAL = target analyte list
TAL Metals = Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Tl, V, Zn

[a] Holding times per SW-846 or method
[b] 14 days to extraction, 40 days from extraction to analysis
[c] 14 days to extraction, 28 days from extraction to analysis
[d] 7 days to extraction, 40 days from extraction to analysis
[e] Method modified for soil analysis

Table C2-2. Sampling Locations, Analysis, and Quality Control Samples

Sample Type	Estimated No. Samples per Location	Sample Interval	Number of Subareas / Locations	Analysis	No. Primary Samples	Estimated No. QC Samples			
						Field Duplicate Samples (FDUP)	Equipment Rinsate Blanks (ERB)	Field Blank (FB)	MS/MSD
Shallow Soil	Three discrete	0-6 in. 6-12 in. 12-24 in.	9	TAL Metals	27	2	2	1 per day	1
				SVOCs[b]	27		2		
				PFAS	6		1		
Soil Borings	Four discrete[a]	0-6 in. 6-12 in. 12-24 in. 84-96 in.	21	TAL Metals	84	5	5		1
				SVOCs[b]	84		5		
				PFAS	17		1		
Surface Water	One discrete	N/A	TBD	TAL Metals (Total)	TBD	TBD	TBD	TBD	TBD
				TAL Metals (Dissolved)	TBD		TBD		
				SVOCs	TBD		TBD		
				1,4-dioxane	TBD		TBD		
				PFAS	TBD		TBD		
				TSS	TBD		TBD		N/A
				TDS	TBD		TBD		N/A
				TOC	TBD		TBD		TBD
				DOC	TBD		TBD		
				pH[c]	TBD		N/A		N/A
				Temperature[c]	TBD		N/A		
				ORP[c]	TBD		N/A		
Sediment	Three discrete	0-6 in. 6-12 in. 12-24 in.	TBD	TAL Metals	TBD	TBD	TBD	TBD	TBD
				SVOCs[b]	TBD		TBD		
				PFAS	TBD		TBD		TBD
				TOC	TBD		N/A		N/A
				Grain Size	TBD		N/A		N/A
				Sulfides	TBD		N/A		N/A
				Nitrate/Nitrite-N	TBD		N/A		N/A
				Carbonates[c]	TBD		N/A		N/A
				pH[c]	TBD		N/A		N/A
				ORP[c]	TBD		N/A		N/A
				CEC[c]	TBD		N/A		N/A

Notes:

CEC = cation exchange capacity
DOC = dissolved organic carbon
MS/MSD = matrix spike/matrix spike duplicate
N/A = not applicable
ORP = oxidation reduction potential
PFAS = per- and polyfluoroalkyl substances
QC = quality control
SVOC = semivolatile organic compound
TAL = Target Analyte List
TAL Metals = Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Tl, V, Zn
TBD = to be determined
TDS = total dissolved solids
TOC = total organic carbon
TSS = total suspended solids

[a] If a layer of ash, brick, and/or glass is encountered, two additional samples will be collected, one within this layer and one from the native material below this layer.
[b] 1,4-dioxane will be reported for 20% of the samples
[c] Field parameters

Table C2-3. Analytes, Method Reporting Limits, and Method Detection Limits[a]

Analyte	Analytical Method	Soil/Sediment		Water	
		MDL	MRL	MDL	MRL
Conventional Chemistry		**mg/kg**		**mg/L**	
Dissolved organic carbon (DOC)	SM 5310D	--	--	0.434	1
Grain size	ASTM D422	--	--	--	--
Nitrate/Nitrite-N	EPA 353.2	0.4	1	--	--
Sulfides	SM 4500-S2 F	11.4	20	--	--
Total dissolved solids (TDS)	SM 2540C	--	--	4	10
Total suspended solids (TSS)	SM 2540D	--	--	4	4
Total organic carbon (TOC)	Lloyd Kahn (soil)/SM 5310D (water)	671	1000	0.434	1
TAL Metals		**mg/kg**		**mg/L**	
Aluminum	SW846 6010C	4.4	10	0.06	0.2
Antimony	SW846 6010C	0.4	15	0.00679	0.02
Arsenic	SW846 6010C	0.4	2	0.00555	0.015
Barium	SW846 6010C	0.11	0.5	0.0007	0.002
Beryllium	SW846 6010C	0.028	0.2	0.0003	0.002
Cadmium	SW846 6010C	0.03	0.2	0.0005	0.002
Calcium	SW846 6010C	3.3	50	0.1	0.5
Chromium	SW846 6010C	0.2	0.5	0.001	0.004
Cobalt	SW846 6010C	0.05	0.5	0.00063	0.004
Copper	SW846 6010C	0.21	1	0.0016	0.01
Iron	SW846 6010C	3.5	10	0.0193	0.05
Lead	SW846 6010C	0.24	1	0.003	0.01
Magnesium	SW846 6010C	0.927	20	0.0434	0.2
Manganese	SW846 6010C	0.032	0.2	0.0004	0.003
Mercury	SW846 7471A(soil)/SW846 7470A (water)	0.0081	0.02	0.00012	0.0002
Nickel	SW846 6010C	0.23	5	0.00126	0.01
Potassium	SW846 6010C	20	30	0.1	0.5
Selenium	SW846 6010C	0.4	4	0.0087	0.025
Silver	SW846 6010C	0.2	0.6	0.0017	0.006
Sodium	SW846 6010C	13	140	0.324	1
Thallium	SW846 6010C	0.3	6	0.0102	0.02
Vanadium	SW846 6010C	0.11	0.5	0.0015	0.005
Zinc	SW846 6010C	0.64	2	0.0015	0.01

Table C2-3. Analytes, Method Reporting Limits, and Method Detection Limits[a]

Analyte	Analytical Method	Soil/Sediment MDL	Soil/Sediment MRL	Water MDL	Water MRL
Semivolatile Organic Compounds (SVOCs)		**µg/kg**		**µg/L**	
Atrazine	SW846 8270D	59	170	0.46	5
Biphenyl	SW846 8270D	25	170	0.653	5
bis(2-chloroisopropyl)ether [2,2'-Oxybis(1-chloropropane)]	SW846 8270D	34	170	0.52	5
1,4-Dioxane	SW846 8270D	55	100	0.1	0.2
2,4,5-Trichlorophenol	SW846 8270D	46	170	0.48	5
2,4,6-Trichlorophenol	SW846 8270D	34	170	0.61	5
2,4-Dichlorophenol	SW846 8270D	18	170	0.51	5
2,4-Dimethylphenol	SW846 8270D	41	170	0.5	5
2,4-Dinitrophenol	SW846 8270D	784	1660	2.22	10
2,4-Dinitrotoluene	SW846 8270D	35	170	0.447	5
2,6-Dinitrotoluene	SW846 8270D	20	170	0.4	5
2-Chloronaphthalene	SW846 8270D	28	170	0.46	5
2-Chlorophenol	SW846 8270D	31	170	0.53	5
2-Methylphenol	SW846 8270D	20	170	0.4	5
2-Methylnaphthalene	SW846 8270D	34	170	0.6	5
2-Nitroaniline	SW846 8270D	25	330	0.42	10
2-Nitrophenol	SW846 8270D	48	170	0.48	5
3,3'-Dichlorobenzidine	SW846 8270D	200	330	0.4	5
3-Nitroaniline	SW846 8270D	47	330	0.48	10
4,6-Dinitro-2-methylphenol	SW846 8270D	170	330	2.2	10
4-Bromophenyl phenyl ether	SW846 8270D	24	170	0.45	5
4-Chloro-3-methylphenol	SW846 8270D	42	170	0.45	5
4-Chloroaniline	SW846 8270D	42	170	0.59	5
4-Chlorophenyl phenyl ether	SW846 8270D	21	170	0.35	5
4-Methylphenol	SW846 8270D	20	330	0.36	10
4-Nitroaniline	SW846 8270D	89	330	0.25	10
4-Nitrophenol	SW846 8270D	119	330	1.52	10
Acenaphthene	SW846 8270D	25	170	0.41	5
Acenaphthylene	SW846 8270D	22	170	0.38	5
Acetophenone	SW846 8270D	23	170	0.54	5
Anthracene	SW846 8270D	42	170	0.28	5
Atrazine	SW846 8270D	59	170	0.46	5
Benzaldehyde	SW846 8270D	135	170	0.267	5
Benzo[a]anthracene	SW846 8270D	17	170	0.36	5
Benzo[a]pyrene	SW846 8270D	25	170	0.47	5
Benzo[b]fluoranthene	SW846 8270D	27	170	0.34	5
Benzo[g,h,i]perylene	SW846 8270D	18	170	0.35	5
Benzo[k]fluoranthene	SW846 8270D	22	170	0.73	5

Table C2-3. Analytes, Method Reporting Limits, and Method Detection Limits[a]

Analyte	Analytical Method	Soil/Sediment		Water	
		MDL	MRL	MDL	MRL
Bis(2-chloroethoxy)methane	SW846 8270D	36	170	0.35	5
Bis(2-chloroethyl)ether	SW846 8270D	22	170	0.4	5
Bis(2-ethylhexyl)phthalate	SW846 8270D	58	170	1.8	5
Butylbenzylphthalate	SW846 8270D	28	170	0.42	5
Caprolactam	SW846 8270D	51	170	2.2	5
Carbazole	SW846 8270D	20	170	0.3	5
Chrysene	SW846 8270D	38	170	0.33	5
Dibenz[a,h]anthracene	SW846 8270D	30	170	0.42	5
Di-n-butylphthalate	SW846 8270D	29	170	0.31	5
Di-n-octylphthalate	SW846 8270D	20	170	0.47	5
Dibenzofuran	SW846 8270D	20	170	0.51	10
Diethylphthalate	SW846 8270D	22	170	0.22	5
Dimethylphthalate	SW846 8270D	20	170	0.36	5
Fluoranthene	SW846 8270D	18	170	0.4	5
Fluorene	SW846 8270D	20	170	0.36	5
Hexachlorobenzene	SW846 8270D	23	170	0.51	5
Hexachlorobutadiene	SW846 8270D	25	170	0.68	5
Hexachlorocyclopentadiene	SW846 8270D	23	170	0.59	5
Hexachloroethane	SW846 8270D	22	170	0.59	5
Indeno[1,2,3-cd]pyrene	SW846 8270D	21	170	0.47	5
Isophorone	SW846 8270D	36	170	0.43	5
N-Nitrosodi-n-propylamine	SW846 8270D	29	170	0.54	5
N-Nitrosodiphenylamine	SW846 8270D	138	170	0.51	5
Naphthalene	SW846 8270D	22	170	0.76	5
Nitrobenzene	SW846 8270D	19	170	0.29	5
Pentachlorophenol	SW846 8270D	170	330	2.2	10
Phenanthrene	SW846 8270D	25	170	0.44	5
Phenol	SW846 8270D	26	170	0.39	5
Pyrene	SW846 8270D	20	170	0.34	5

Table C2-3. Analytes, Method Reporting Limits, and Method Detection Limits[a]

Analyte	Analytical Method	Soil/Sediment		Water	
		MDL	MRL	MDL	MRL
Per- and polyfluoroalkyl substances (PFAS)		**µg/kg**		**ng/L**	
Perfluorobutanoic acid (PFBA)	EPA 537.1M	0.19	0.5	1.13	5
Perfluoropentanoic acid (PFPeA)	EPA 537.1M	0.018	0.2	1.08	2
Perfluorohexanoic acid (PFHxA)	EPA 537.1M	0.024	0.2	0.83	2
Perfluoroheptanoic acid (PFHpA)	EPA 537.1M	0.023	0.2	0.46	2
Perfluorooctanoic acid (PFOA)	EPA 537.1M	0.014	0.2	0.98	2
Perfluorononanoic acid (PFNA)	EPA 537.1M	0.02	0.2	0.58	2
Perfluorodecanoic acid (PFDA)	EPA 537.1M	0.021	0.2	0.46	2
Perfluoroundecanoic acid (PFUnA)	EPA 537.1M	0.024	0.2	0.73	2
Perfluorododecanoic acid (PFDoA)	EPA 537.1M	0.015	0.2	0.46	2
Perfluorotridecanoic acid (PFTriA)	EPA 537.1M	0.013	0.2	0.43	2
Perfluorotetradecanoic acid (PFTeA)	EPA 537.1M	0.019	0.2	0.59	2
Perfluorobutanesulfonic acid (PFBS)	EPA 537.1M	0.0088	0.2	0.63	2
Perfluorohexanesulfonic acid (PFHxS)	EPA 537.1M	0.015	0.2	0.67	2
Perfluoroheptanesulfonic Acid (PFHpS)	EPA 537.1M	0.015	0.2	0.39	2
Perfluorooctanesulfonic acid (PFOS)	EPA 537.1M	0.067	0.2	0.87	2
Perfluorodecanesulfonic acid (PFDS)	EPA 537.1M	0.019	0.2	0.48	2
Perfluorooctanesulfonamide (FOSA)	EPA 537.1M	0.0088	0.2	0.57	2
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	EPA 537.1M	0.034	2	0.79	5
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	EPA 537.1M	0.03	2	0.93	5
6:2 FTS	EPA 537.1M	0.022	2	0.72	5
8:2 FTS	EPA 537.1M	0.029	2	0.66	2
1,4-Dioxane		**µg/kg**		**µg/L**	
1,4-Dioxane	SW846 8270D SIM ID	--	--	0.1	0.2

Notes:
-- = not applicable
ASTM = American Society of Testing and Materials
EPA = U.S. Environmental Protection Agency
ID = isotope dilution
MDL = method detection limit
MRL = method reporting limit
SIM = selected ion monitoring
SM = Standard Method
SW846 = Test Methods for Evaluating Solid Waste: Physical/Chemical Methods
TAL = Target Analyte List
TBD = to be determined

[a]The MDLs and MRLs reflect TestAmerica (TA) Buffalo limits at the time the QAPP was prepared. MDLs and MRLs reported may differ from these limits. MDLs and MRLs may change over the span of the project. If other TA network laboratories are utilized for the project; MDLs and MRLs may differ.

ATTACHMENT 1

LABORATORY QUALITY ASSURANCE MANUAL

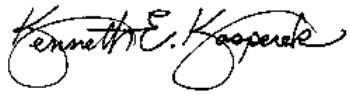
Cover Page:

Quality Assurance Manual

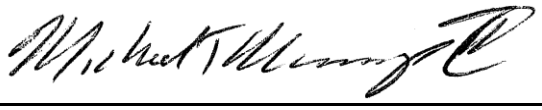
Eurofins TestAmerica Buffalo
10 Hazelwood Drive
Amherst, New York 14228
716.504.9800
716.691.7991

www.testamericainc.com

Title Page:
Quality Assurance Manual
Approval Signatures



Laboratory Director – Kene' Kasperek — 3/21/2018 Date

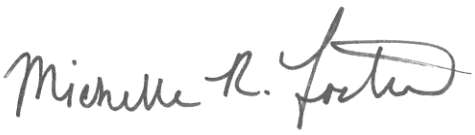


Quality Assurance Manager - Michael Mosscrop — 3/21/2018 Date

Inorganics Operations Manager – Jennifer Pierce — 3/21/2018 Date

Organic Operations Manager – Gary Rudz — 3/21/2018 Date

Organic Preparation Manager – Vikrambhai Patel — 3/21/2018 Date



Wet Chemistry Manager – Michelle Foster — 3/21/2018 Date

Metals Manager – Jason Kacalski — 3/21/2018 Date

GC/MS Semivolatiles – Michelle Page — 3/21/2018 Date

GC/MS Volatiles – Leah Hill — 3/21/2018 Date

Facilities Manager – Ken Kinecki — 3/21/2018 Date

SECTION 2

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-I-P-002	Electronic Reporting and Signature Policy

CA-L-P-002	Contract Compliance Policy
CW-L-S-004	Subcontracting Procedures
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CA-C-S-001	Work Sharing Process

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-PM-008	Massachusetts DEP Notification Procedures
BF-QA-001	Determination of Method Detection Limits
BF-QA-002	Quality Control Limits
BF-QA-003	Procedure for Writing, Reviewing and Revising Controlled Documents
BF-QA-004	Laboratory Personnel Training
BF-QA-005	Preventative and Corrective Action
BF-QA-006	Data Quality Review
BF-SR-001	Cooler Shipping - Bottle Kits and Samples
BF-SR-002	Receipt of Analytical Samples

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

SECTION 3

INTRODUCTION, SCOPE AND APPLICABILITY

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

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

The QAM has been prepared to assure compliance with 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards, The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E) In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality 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:

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- 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; Final Update V, August 2015
- 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 Order 414.1D, Quality Assurance, Aril, 25, 2011.*
- 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 and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director 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 and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive VP Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Buffalo is presented in Figure 4-1.

4.2 Roles and Responsibilities

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

4.2.1 Additional Requirements for Laboratories

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

4.2.2 Laboratory Director

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

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

Specific responsibilities include, but are not limited to:

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

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

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

4.2.3 Quality Assurance (QA) Manager or Designee

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

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

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

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems, data authenticity and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a subset of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, evaluate manual calculations, format, holding time, sensibility and completeness of the project file contents.
- 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.

4.2.4 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

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

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

4.2.5 Operations Manager

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

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

4.2.6 Department Managers

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

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

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

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

4.2.7 Hazardous Waste Coordinator

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

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

4.2.8 Environmental Health & Safety Coordinator

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

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

4.2.9 Laboratory Analysts

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

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

- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

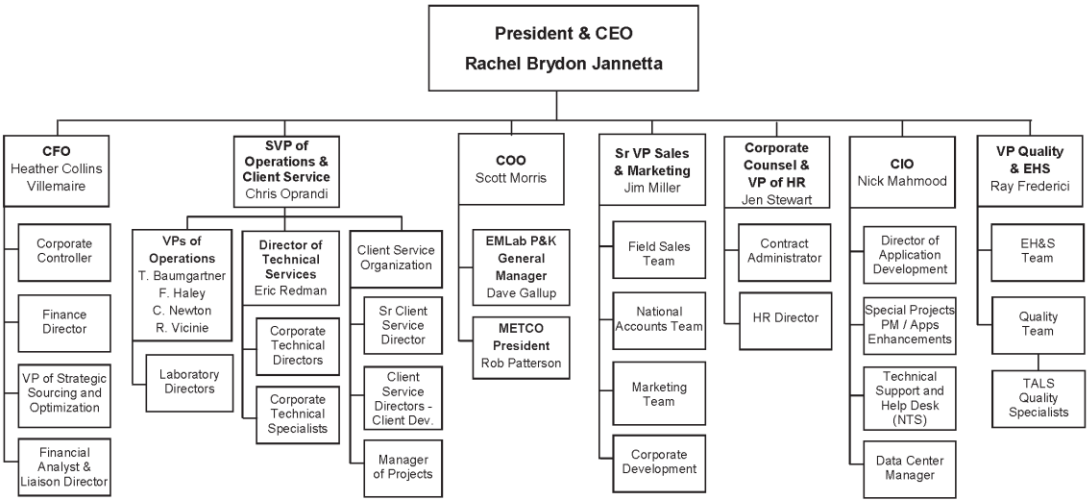
4.3 DEPUTIES

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

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

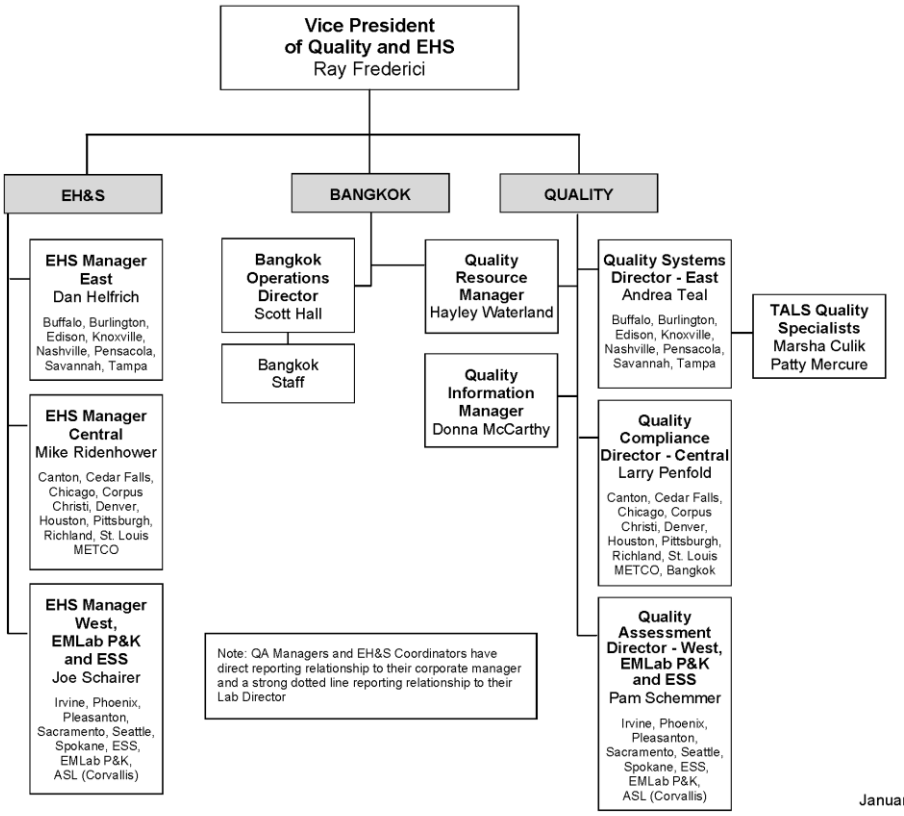
Figure 4-1.
Corporate and Laboratory Organization Charts

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

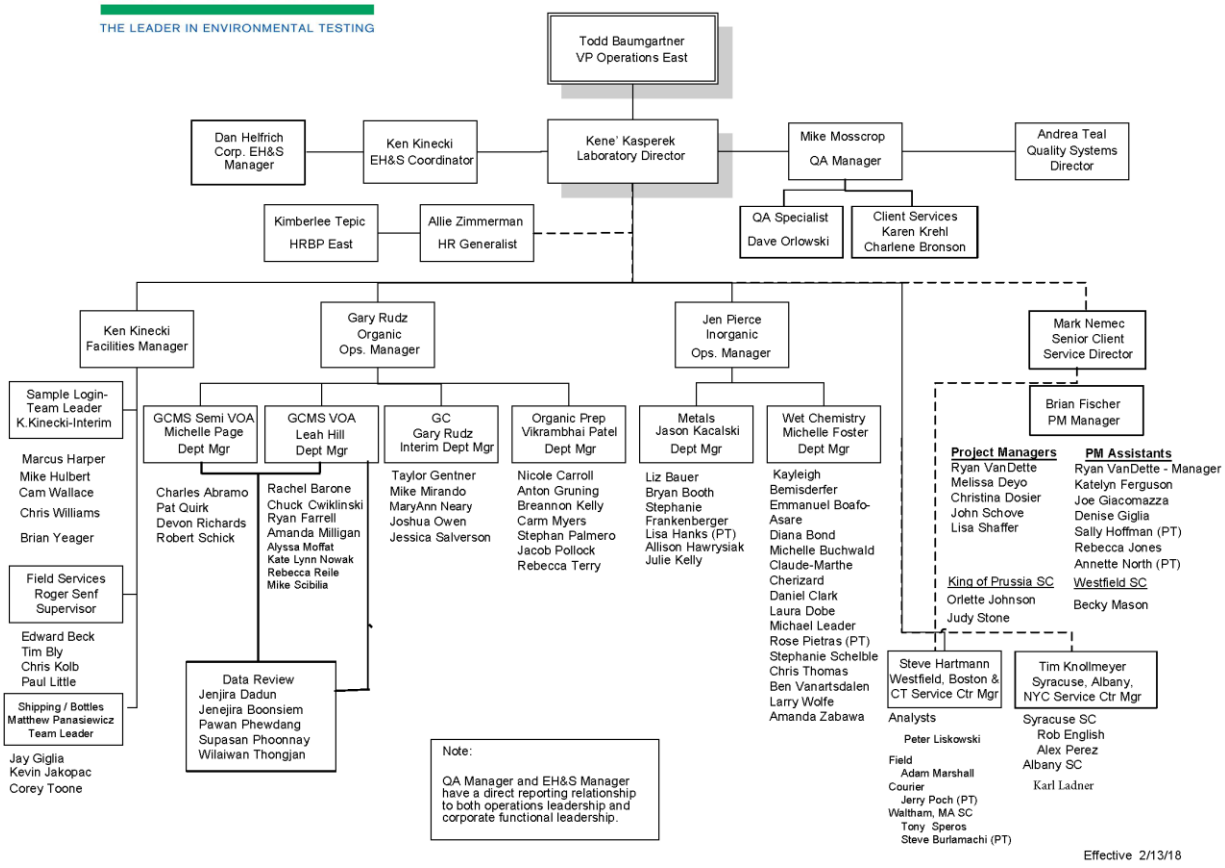
Quality, EHS, Bangkok



January 3, 2018

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Buffalo Laboratory Organization



Note: Organizational Charts are current at the date of publication of this manual. Updated charts may be obtained by contacting the TestAmerica Buffalo Quality Department.

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-Q-S-005).

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

5.3 QUALITY SYSTEM DOCUMENTATION

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

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

5.3.1 Order of Precedence

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

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

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

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

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

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

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

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

5.4.1 Precision

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

5.4.2 Accuracy

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

A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

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

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

5.4.4 Comparability

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

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

5.4.5 Completeness

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

5.4.6 Selectivity

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

5.4.7 Sensitivity

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

5.5 CRITERIA FOR QUALITY INDICATORS

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

5.6 STATISTICAL QUALITY CONTROL

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

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

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

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

5.6.1 QC Charts

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

5.7 QUALITY SYSTEM METRICS

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

SECTION 6

DOCUMENT CONTROL

6.1 OVERVIEW

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

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

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

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

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

6.2 DOCUMENT APPROVAL AND ISSUE

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

Controlled documents are authorized by the QA Department. In order to develop a new document, a Department Manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version

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

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

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

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory public storage server (L: drive) or through the TALS File Share menu within the LIMS, and is available to all personnel.

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

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

6.4 OBSOLETE DOCUMENTS

When revisions are implemented for an SOP, form or work instruction, the previous document becomes obsolete and is archived. 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 destroyed. At least one copy of the obsolete document is archived according to SOPs No. BF-GP-015 and BF-QA-003. All archived SOPs, manuals, forms or work instructions are considered obsolete.

SECTION 7

SERVICE TO THE CLIENT

7.1 OVERVIEW

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

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

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

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

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

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

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

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

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

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

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

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

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

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contact Administrator
- VP of Operations
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts. The Project Managers at the TestAmerica Buffalo facility also maintains copies of these documents.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal a PM is assigned to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers/Designees are available to discuss any technical questions or concerns that the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOP’s on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (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), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, may require notification prior to placing such work**.**

Approval may be documented through reference in a quote / contract or e-mail correspondence.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Account Executive (AE) or Client Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory.
- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor. 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.
- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

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).

8.2.1 When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager/Designee begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.3 OVERSIGHT AND REPORTING

8.3.1 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Corporate 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 CSO personnel 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 CSO Personnel, Laboratory Directors/Managers, QA Managers and Sales Personnel.

Prior to initially 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 within the project records.

8.3.2 For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

Note: Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company TotalAccess Database.

8.3.3 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 be available in TALS for 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 use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9

PURCHASING SERVICES AND SUPPLIES

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment, consumables 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. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica Sharepoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Purchase requisitions are placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

9.3.2 Receiving

It is the responsibility of the purchasing manager/designee to receive the shipment. It is the responsibility of the department that ordered the materials to document the date the materials were 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. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager/designee verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot not be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.

- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- umho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{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 Manager or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. DOC No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the

services are approved by the Department Managers, Operations Manager and/or Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP BF-GP-002,. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as NIST thermometers, weight sets, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurements & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (available on the intranet site).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Manager are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS

10.1 OVERVIEW

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, e.g., communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in the laboratory SOP related Corrective Action (BF-QA-005).

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to BF-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for resolution. The department manager may elect to discuss it with the Technical Manager, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's non-conformance and corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director, Technical Manager, Operations Manager or QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with the analytical method requirements and the reason.

11.2 RESPONSIBILITIES AND AUTHORITIES

Under certain circumstances the Laboratory Director, the Technical Manager, the Operations Manager or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's non-conformance and corrective action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review checklists as appropriate**.** Any

impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Manager, and QA Manager. Suspected misrepresentation issues may also be reported to any member of the corporate staff as identified in Ethics Policy, CW-L-P-004. 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), (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

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-Q-S-005.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc…). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12

CORRECTIVE ACTION

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memo (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Project Management concerns regarding specific analytical results
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.

- Internal and External Audit Findings
- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. A NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Operations Manager, Technical Manager, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into the Laboratory Information Management System (LIMS) and each CAR is entered into the Incident and Corrective Action Tracker (iCAT) database for tracking and trending purposes for review to aid in ensuring that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local spreadsheet database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document

upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.

- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.
- Also refer to Section 15.1.4, Special Audits)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of a NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, work instructions**,** QAM Sections 19 and 20. All corrective actions are reviewed monthly at a minimum by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example – iCAT Corrective Action Notice

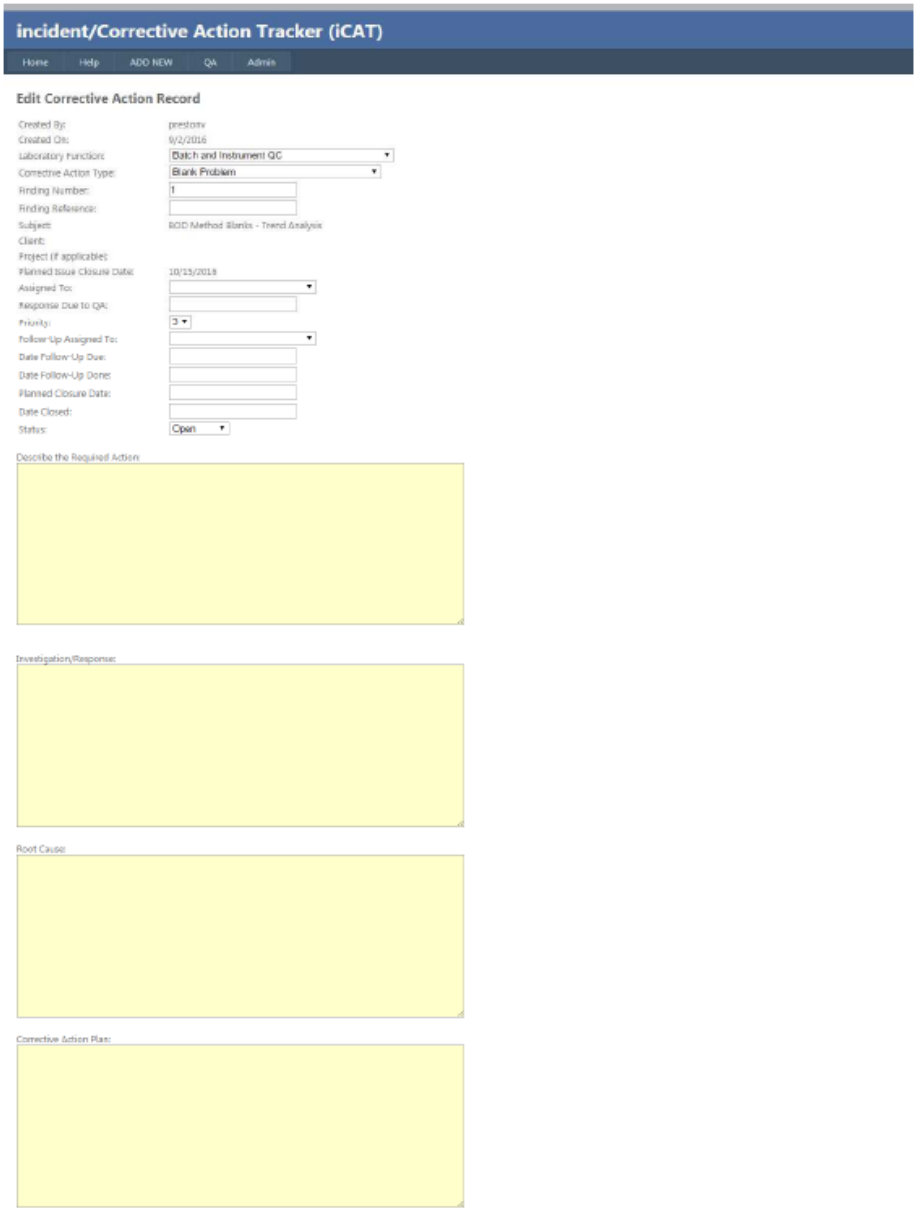


Table 12-1. Example – General Corrective Action Procedures

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 Manager, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors *(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)*	- SOP CW-Q-S-005, Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-Q-S-005 or lab SOP BF-QA-005
Client Complaints *(Project Managers, Lab Director, Sales and Marketing, QA Manager)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) *(QA Manager, Lab Director, Operations Manager Department Managers)*	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation *(EH&S Coordinator, Lab Director, Operations Manager, Department Manager)*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through EH&S office.

Note: 1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the

ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and the other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

.

SECTION 13.0

PREVENTIVE ACTION / IMPROVEMENT

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's Corrective Action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further

occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

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

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvment.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review

13.1.2 Any Preventive Actions/Process Improvements undertaken or attempted shall be taken into account during the Annual Management Systems Review (Section 17). A highly detailed report is not required; however a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

SECTION 14.0

CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention, specifies 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. . More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. 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 Laboratory Director and the QA Department while electronic technical records are maintained by the IT Administrator.

14.1.1 All records are stored and retained according to BF-GP-015 and in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss.. All records shall be protected against fire, theft, loss, environmental deterioration and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log.

If records are archived off-site they are to be stored in a secure location where a record is maintained of any entry into the storage facility. Records are maintained for a minimum of five years unless other wise 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.

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 - Published Methods	Indefinitely
QA Records	- Certifications - Method & Software Validation / Verification Data	Indefinitely
	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - 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)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP / SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	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	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard & sample), Standard & Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

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	10 years (lab reports and raw data) 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
OSHA	30 years

[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 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (any records stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the Job Number in TALS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The

records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

14.2.2 Observations, data and calculations are recorded real-time.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries.
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

14.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

14.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

14.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

14.5.4 The laboratory has a record management system (also known as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in LIMS and logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.

14.5.5 Records are considered archived when noted as such in the records management system (also known as document control). Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned.

14.5.6 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.7 Records Disposal

14.5.7.1 Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

14.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read. If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required**.**

SECTION 15

AUDITS

15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. 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 QA Technical Data Audits SOP Compliance Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	QA Methods Audits Frequency: All methods are reviewed annually. 50% of methods receive a QA Technical Audit 50% of methods receive a SOP Method Compliance Audit
Special	QA Department or Designee	Surveillance or spot checks performed as needed to monitor specific issues
Performance Testing	Coordinated by Corporate QA	Two successful per year for each TNI - NELAP field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These 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. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil, and Air.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Director prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Manager, Operations Manager, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.

- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes.

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The TestAmerica Corporate Internal Investigations 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 President and CEO, COO, Technical & Operations Support, VP of Client and Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located in the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers/Department Managers – **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 – Comprehensive 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; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; May 18, 2012 and/or August 28 , 2017 (depending on state implementation timelines).*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*

- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th / 21st/ 22nd/on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)* ***(DW labs only)***
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *New York State DEC Analytical Services Protocol, 2005*
- *New York State DOH Methods Manual*
- *Massachusetts Contingency Plan 310 CMR 40, April 25, 2014*
- *Connecticut Reasonable Confidence Protocol, July 2006*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability (BF-QA-004) is performed whenever there is a significant change in instrument type (e.g., new instrumentation), method or personnel.

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager/Designee and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified

as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 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 can be differentiated from blanks. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, regulations, 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 several days to provide a more realistic MDL. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used. Where required by 40 CFR Part 136, Appendix B, continuing MDLs will be calculated from a minimum of 7 spiked replicates analyzed quarterly and compared to statistical method blank data to determine the final updated MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. BF-QA-001 for details on the laboratory's MDL process.

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

19.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirement.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory's SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, and specific electrode response factors.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ±0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as "reanalysis") may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 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 Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are 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 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review –The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

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

19.14.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. When complete, the report is issued to the client.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines**.**

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1.
Example - Demonstration of Capability Documentation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

BF-QA-DOC-004
DOC Cert. Statement
Rev. 3 9/28/2016

TESTAMERICA LABORATORIES, INC.

DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee Name (print): _____

Method Number: _____ Matrix (circle): water / soil / air

Parameters or Analytes: _____

Date Submitted: _____

Initial Demonstration of Capability:

SOP Number: _____Revision # _____ Date Read_____

Trained By (print name): _____

Date training began: _____

Date training completed: _____

Continued Demonstration of Capability:

SOP Number: _____Revision # _____ Date Read_____

Demonstration of Capability Reviewed and Analyst Authorized to Perform Method:

_____ _____ _____
Department Manager/Designee Signature Date

_____ _____ _____
QA Manager/Designee Signature Date

SECTION 20

EQUIPMENT (AND CALIBRATIONS)

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the

replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrumentation records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses

20.2.6 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

Laboratory SOPs BF-GP-001,"Calibration of Autopipettes and 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 ± 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.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual. The IR thermometers are verified daily and calibrated quarterly. Digital probes and thermocouples are calibrated

quarterly. Disposable thermometers are discarded upon expiration and replaced with newly purchased thermometers.

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 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 is ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration 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 2009 TNI Std. EL-V1M4, section 1.7.1. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met i.e., RPD, per NELAC (2003) Standard, Section 5.5.5.10 and 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification 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, rev. 11-3-2017

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS Instrumentation	Agilent	5975	US83110163	2013	good
GC/MS Instrumentation	Agilent	5973	US02450141	2012	good
GC/MS Instrumentation	Agilent	5975	US83130241	2013	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	US21854062	2003	good
GC/MS Instrumentation	Agilent	5973	US30965634	2003	good
GC/MS Instrumentation	Agilent	5973	US03965692	2003	good
GC/MS Instrumentation	Agilent	5973	US05605976	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	680S10101807	2013	good
GC Instrumentation	Perkin Elmer	Clarus 600 dual FID	665S10020401	2012	good
GC Instrumentation	Agilent	6890 dual uECD	CN10839003	2005	good
GC Instrumentation	Agilent	6890 dual uECD	CN10833020	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

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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	580II FID/FID	3336A53729	1994	good
GC Instrumentation	Hewlett Packard	580II FID/FID	3336A53728	1994	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3310A47661	1993	good
GC Instrumentation	Hewlett Packard	5890II dual ECD	3336A53325	1993	good
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
Metals Instrumentation	Environmental Express	AutoBlock Plus	AB4001-1213-042	2013	good
Water Quality Instrumentation	ManTech	PC Titrator	PCM-PSDT/CA	2015	good
Water Quality Instrumentation	Metrohm	IC Model 881	4111	2013	good

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Quality Instrumentation	Konelab	Aqua20	SEA032	2009	good
Water Quality Instrumentation	Flash Point Analyzer	HFP 339	73390092	2007	good
Water Quality Instrumentation	Flash Point Analyzer	Optiflash 104002	Herzog PAC 000334	2015	good
Water Quality Instrumentation	OI	Carbon Analyzer Model 1030	A549730578	2006	good
Water Quality Instrumentation	OI	Carbon Analyzer Model 1030	E616730030	2006	good
Water Quality Instrumentation	OI	Carbon Analyzer Model 1030	P410730479	2003	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	Metrohm	881 Compact IC Pro	36756	2014	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	Mantech	BOD Autoanalyzer	MS-1LO-157	2004	good
Water Quality Instrumentation	Mantech	BOD Autoanalyzer	MT-0**B**4-215	2015	good
Water Quality Instrumentation	Mantech	PC Titrator	MS-OK2-607	2003	good
Water Quality Instrumentation	HACH	Spectrophotometer #DR/2500	30200004886	2003	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	2060196	2002	good
Water Quality Instrumentation	Spectronic	Genesis 4001/4	3SGC199091	2000	good
Water Quality Instrumentation	Lachat	Quickchem 8000 Autoanalyzer	A83000-1527	2000	good
Water Quality Instrumentation	Lachat	Quickchem 8500 Autoanalyzer	40300001665	2014	good

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Quality Instrumentation	Lachat	Quickchem 8500 Autoanalyzer	11060001336	2013	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	99010157	1999	good
Water Quality Instrumentation	Dionex	Ion Chromatograph #DX-120	99110569	1999	good
Water Quality Instrumentation	BOD chamber		Revco	1994	good
Sample Preparation Equipment	CEM	Microwave MARS	MD3978	2013	good
Sample Preparation Equipment	Gilson	Fractionator Model GX-274	40579	2013	good
Sample Preparation Equipment	TurboVap	II	TV0529N12427	2006	good
Sample Preparation Equipment	TurboVap	II	TV0529N12428	2006	good
Sample Preparation Equipment	TurboVap	II	TV9445N5816	1996	good
Sample Preparation Equipment	TurboVap	II	TV9427N4133	1996	good
Sample Preparation Equipment	TurboVap	II	TV944N5819	1996	good
Sample Preparation Equipment	TurboVap	II	TV944N5820	1996	good
Sample Preparation Equipment	TurboVap	II	TV0024N9623	2000	good
Sample Preparation Equipment	TurboVap	II	TV0022N9604	2000	good
Sample Preparation Equipment	TurboVap	II	TV0312N11592	2003	good
Sample Preparation Equipment	TurboVap	II	TV0312N11591	2003	good
Sample Preparation Equipment	Heat Systems	Sonicator #XL-2020	G1647/C5659	1994	good

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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
Sample Preparation Equipment	Organomation	Rot-X-Tractor	169902	1999	good
Sample Preparation Equipment	Organomation	Rot-X-Tractor	16907	1999	good
Sample Preparation Equipment	Organomation	Rot-X-Tractor	16913	1999	good

Note: The Equipment List is current at the date of publication of this manual. An updated list may be obtained by contacting the TestAmerica Buffalo Quality Department.

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 Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Glass wool replacement Check system for gas leaks with SNOOP Check for loose/frayed power wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required As required W/cylinder change as required As Required As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples and solvents Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
Centrifuge	Check brushes and bearings	Every 6 months or as needed

Table 20-3.

Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using "S" NIST traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	± 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	± 2.0°C	Replace
Minimum-Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 2.0°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	± 2.0°C	Repair/replace
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 2.0°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 Manager.

SECTION 21

MEASUREMENT TRACEABILITY

21.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. For certain programs Microsyringes are verified semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g. bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory accreditation Cooperation) or APLAC (Asia – Pacific Laboratory Accreditation Cooperation)..A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to ***TestAmerica Buffalo*** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, “Standard Traceability and Preparation” and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory’s LIMS system or 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 in the electronic database within the LIMS:

- Standard ID
- Description of Standard
- Department
- Preparer’s name
- Final volume and number of vials prepared
- Solvent type and lot number

- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID from LIMS.
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the LIMS system.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

SECTION 22.0

SAMPLING

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

BF-FS-001	Chain of Custody Documentation
BF-FS-003	Groundwater Sampling Field Data Collection
BF-FS-004	Equipment Decontamination
BF-FS-005	Groundwater/Surface Water Sampling
BF-FS-006	Calibration of Field Meter
BF-FS-007	Low Flow Sampling Procedures
BF-FS-008	Surface and Subsurface Soil/Sediment Sampling

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory online.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times, this info is in the SOP or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, "Sample Homogenization and Subsampling".

SECTION 23

HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The shipping documents are retained with the project files.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

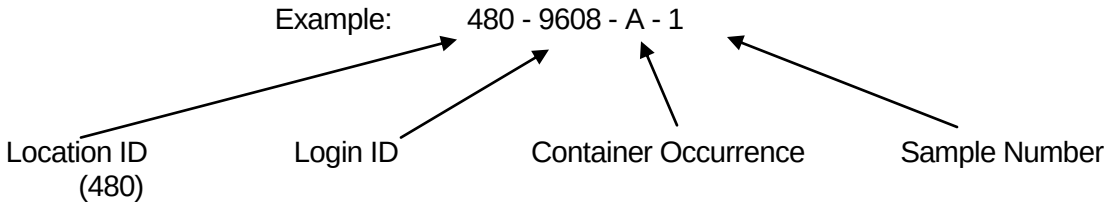
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Login Form – and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Buffalo Laboratory (Location 480). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);

- every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.;
- The project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks described in Section 23.1.1.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. BF-SR-002.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis

the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel.

All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses (see Note), a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will analyze the trip blanks that were supplied.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record should be completed.

Figure 23-1.

Example: Chain of Custody (COC)

Chain of Custody Record XXXXXX

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Laboratories, Inc.
TAL-8210

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

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Company Name:	Tel/Fax:	Lab Contact:	Carrier:	_____ of _____ COCs
Address:	Analysis Turnaround Time			Sampler:
City/State/Zip:	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
Phone:	TAT if different from Below _____			Walk-in Client:
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:

Figure 23-2. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.***
   - **Information must be legible**

2) Every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.

3) Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

4) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

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

6) Samples must be preserved according to the requirements of the requested analytical method. See lab Sampling Guide.

   Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

   - Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

   - For Volatile Organic analyses in drinking water (Method 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
     - 1. Test for residual chlorine in the field prior to sampling.
       - If no chlorine is present, the samples are to be preserved using HCl as usual.
       - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
     - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.

   - **FOR WATER SAMPLES TESTED FOR CYANIDE – for NPDES samples by Standard Methods or EPA 335**
     - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

       - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements

or the laboratory can analyze the samples as delivered and qualify the results in the final report.

- It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

- The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

7) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.

- Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for “field” analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.

8) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.

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

10) Recommendations for packing samples for shipment.

- Pack samples in Ice rather than “Blue” ice packs.

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

- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

- Fill extra cooler space with bubble wrap.

Figure 23-3.
Example: Cooler Receipt Form (Optional)

BF-SC-LF-003
Rev.2 8/28/2017

SAMPLE LOGIN

Project_____Event _____

Analysis Groups_____

TAT ____ # **SAMPLES**: _____ **TRIP BLANK? Y/N** ___ **#/date** _____

Custody Seal Intact Y/N NONE **Rad Check <0.02 mR/hr Y/N**

Residual Chlorine Check Y/N/ NA **Pres Checked Y/N/NA**

Workshare/Subcontract Y/N Lab_____ _____ **SO/ICOC #**_____

Received out of hold: Samples _____**Analysis**_____

Checklist/NCM's_____

Temperature(s) **#of coolers**_____ **IR Gun** **1** **2** **3**

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. (TNI)
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). In order to meet this requirement, TestAmerica Buffalo spikes with the Corporate Standard Standards primary mix for each analysis. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.4.1.5.1 For methods that have 1-10 target analytes, spike all components.

24.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-5. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 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. This process is outlined in BF-QA-002.

24.6.4.1 The control limits are maintained in the laboratory LIMS system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMS system and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

24.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples.

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 19.

24.7.7 The laboratories sample acceptance policy is included in Section 23.

SECTION 25.0

REPORTING RESULTS

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. A variety of report formats are available to meet specific needs. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

The laboratory complies with any state reporting requirements. An example is located in BF-PM-008 – Massachusetts DEP Notification Procedures.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) with a "sample results" column header.

25.2.2 Each report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Laboratory Practical quantitation limits or client reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the COC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

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

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

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When NELAP accreditation is required, the lab shall certify that the test results meet all requirements of NELAP or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

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

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

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g, partial report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as an addendum to the report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 Certification Summary report, where required, will document that unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

25.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica Buffalo offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data)
- 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. Procedures used to ensure client confidentiality are outlined in Section 26.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD . TestAmerica Buffalo offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document, include cover letter.

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

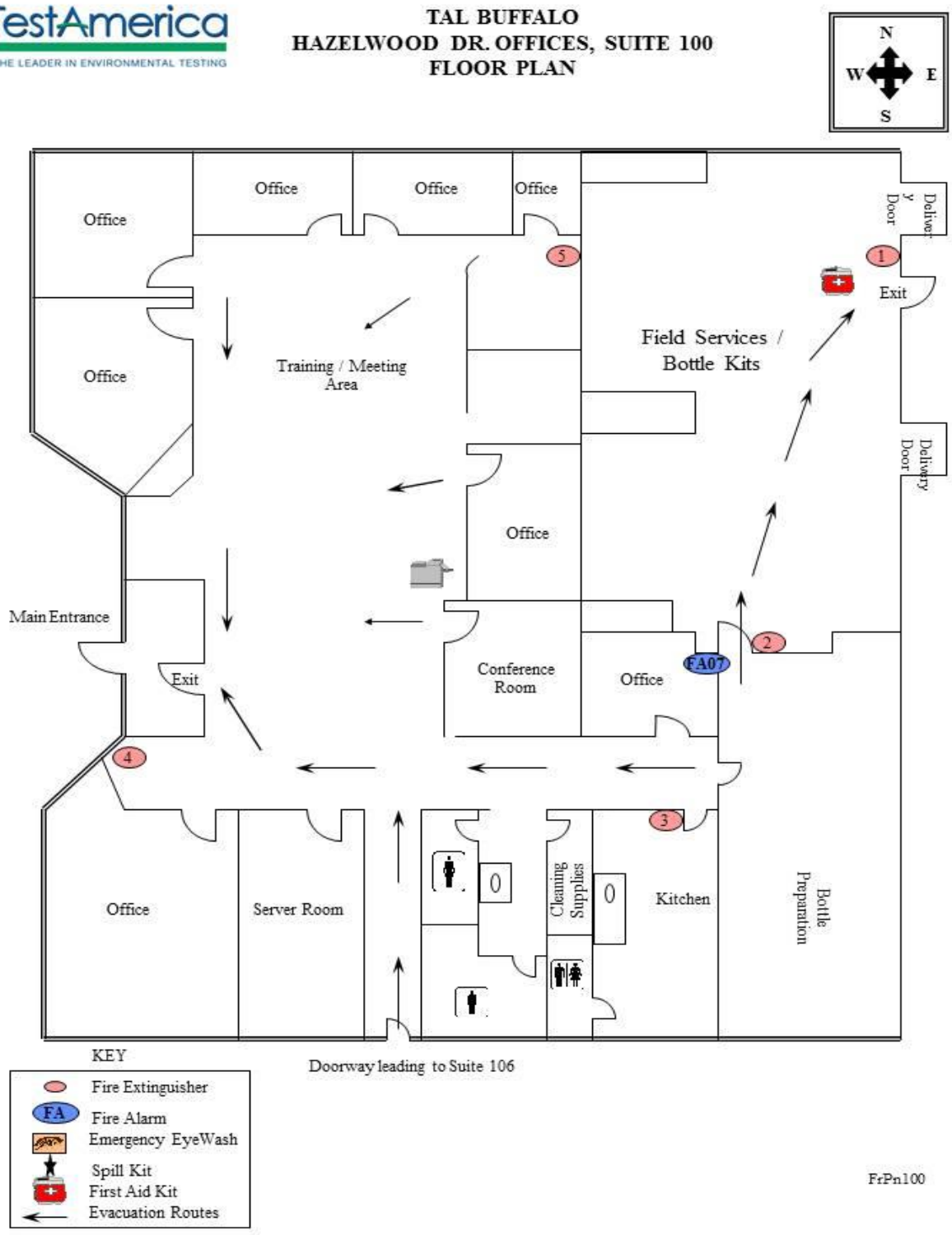
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- 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

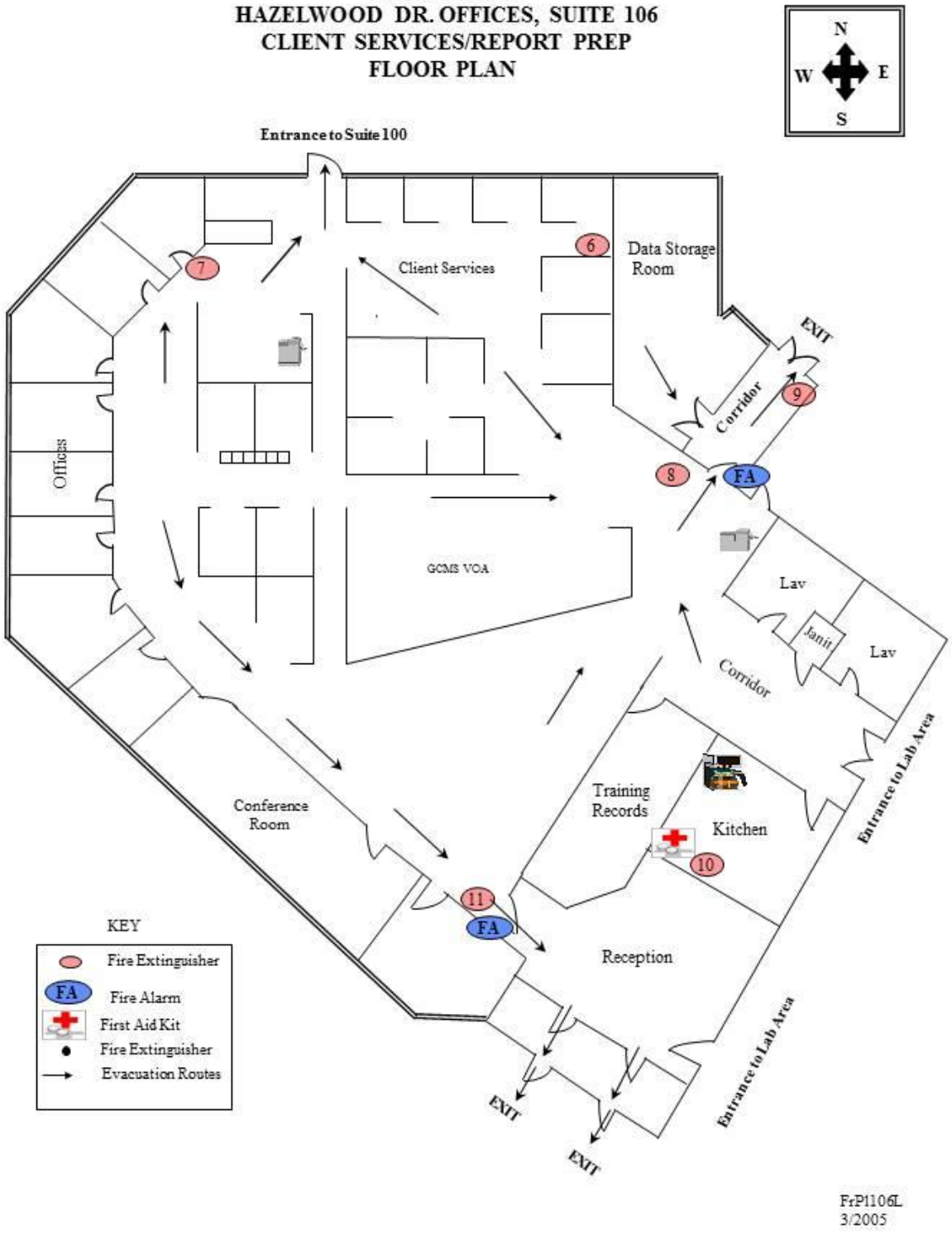
TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

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



FrP1106L
3/2005

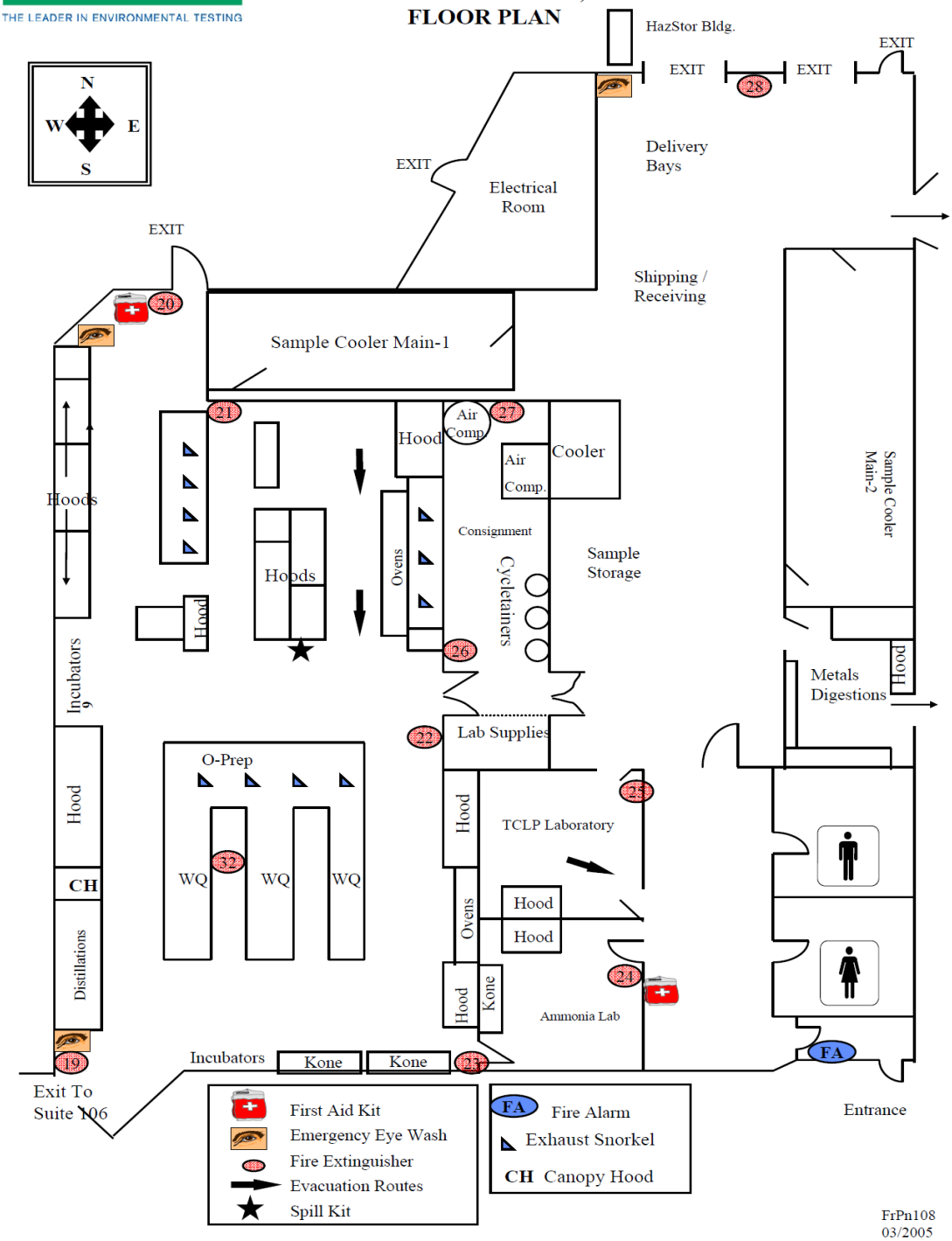
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

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

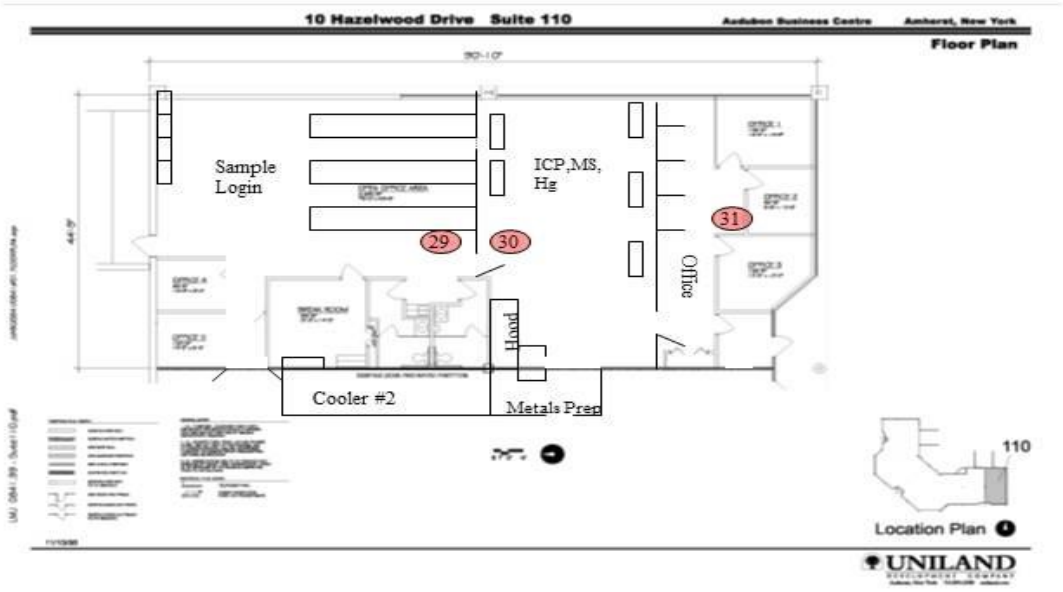
FrPn106r
03/2005

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
Sample Login
ICP,MS, Hg
29
30
31
Office
Hood
Cooler #2
Metals Prep
110
Location Plan
UNILAND

Appendix 2. Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

Accrediting Authority: The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed

together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI).

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (TNI)

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management,

operation or products. TNI 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)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

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), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity 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. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (TNI)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

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. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or and which is accepted as the method for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS-ICP/Mass Spectrometry

ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
TNI – The NELAC Institute
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Buffalo maintains 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:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Buffalo	Federal	USDA	P330-11-00386	02/06/2021
TestAmerica Buffalo	NELAP	Florida	E87672	06/30/2018
TestAmerica Buffalo	NELAP	Illinois	200003	09/30/2018
TestAmerica Buffalo	NELAP	Kansas	E-10187	01/31/2019
TestAmerica Buffalo	NELAP	Louisiana	02031	06/30/2018
TestAmerica Buffalo	NELAP	Minnesota	036-999-337	12/31/2018
TestAmerica Buffalo	NELAP	New Hampshire	2337	11/17/2018
TestAmerica Buffalo	NELAP	New Hampshire	2973	09/11/2018
TestAmerica Buffalo	NELAP	New Jersey	NY455	06/30/2018
TestAmerica Buffalo	NELAP	New York	10026	03/31/2018
TestAmerica Buffalo	NELAP	Oregon	NY200003	06/09/2018
TestAmerica Buffalo	NELAP	Pennsylvania	68-00281	07/31/2018
TestAmerica Buffalo	NELAP	Texas	T104704412-15-6	07/31/2018
TestAmerica Buffalo	NELAP	Virginia	460185	09/14/2018
TestAmerica Buffalo	State Program	Arkansas DEQ	88-0686	07/06/2018
TestAmerica Buffalo	State Program	California	2931	04/01/2018
TestAmerica Buffalo	State Program	Connecticut	PH-0568	09/30/2018
TestAmerica Buffalo	State Program	Georgia	10026 (NY)	03/31/2018
TestAmerica Buffalo	State Program	Georgia	956	03/31/2018
TestAmerica Buffalo	State Program	Iowa	374	03/01/2019
TestAmerica Buffalo	State Program	Kentucky (DW)	90029	12/31/2018
TestAmerica Buffalo	State Program	Kentucky (UST)	30	03/31/2018
TestAmerica Buffalo	State Program	Kentucky (WW)	90029	12/31/2018
TestAmerica Buffalo	State Program	Maine	NY00044	12/04/2018
TestAmerica Buffalo	State Program	Maryland	294	03/31/2018
TestAmerica Buffalo	State Program	Massachusetts	M-NY044	06/30/2018
TestAmerica Buffalo	State Program	Michigan	9937	03/31/2018
TestAmerica Buffalo	State Program	North Dakota	R-176	03/31/2018
TestAmerica Buffalo	State Program	Oklahoma	9421	08/31/2018
TestAmerica Buffalo	State Program	Rhode Island	LAO00328	12/30/2018
TestAmerica Buffalo	State Program	Tennessee	TN02970	03/31/2018
TestAmerica Buffalo	State Program	Washington	C784	02/10/2019
TestAmerica Buffalo	State Program	Wisconsin	998310390	08/31/2018

Page 1 of 1 3/21/18

* Certification Valid - Laboratory is Pending Renewal with the Program Authority

For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

Title: Per- and Polyfluorinated Substances (PFAS) in Potable Water

[Method ISO 25101:2009]

Approval Signatures:

Don Dawicki
Laboratory Director

Kristine Dusablon
Quality Assurance Manager

Matthew Kirk
Operations Mgr./EHS Coordinator

Mark Fausel
Department Supervisor

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

1.0 Scope and Application

This SOP describes the laboratory procedure for the preparation and analysis of per- and polyfluorinated substances using liquid chromatography/tandem mass spectrometry (LC/MS/MS).

1.1 Analytes, Matrices, and Reporting Limits

This procedure applies to potable water samples only.

The list of target compounds that may be determined from this procedure is provided below. Table 1 presents the compounds along with their associated reporting limits (RL).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
*Perfluoro-n-butanoic acid	PFBA	375-22-4
*Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
*Perfluoro-n-hexanoic acid	PFHxA	307-24-4
*Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
*Perfluoro-n-nonanoic acid	PFNA	375-95-1
*Perfluoro-n-decanoic acid	PFDA	335-76-2
*Perfluoro-n-undecanoic acid	PFUnA	2058-94-8
*Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
*Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
*Perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7
Perfluorinated sulfonic acids (PFSAs)		
*Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
*Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-4
*Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
*Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
*Perfluoro-1-nonanesulfonic acid	PFNS	68259-12-1
*Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluorinated sulfonamides (FOSA)		
*Perfluoro-1-octanesulfonamide	FOSA	754-91-6
Perfluorinated sulfonamidoacetic acids (FOSAA)		
*N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
*N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
*1H,1H,2H,2H-perfluorohexanesulfonic acid *(4:2)*	4:2 FTS	757124-72-4
*1H,1H,2H,2H-perfluorooctanesulfonic acid *(6:2)*	6:2 FTS	27619-97-2
*1H,1H,2H,2H-perfluorodecanesulfonic acid *(8:2)*	8:2 FTS	39108-34-4
Fluorinated Replacement Chemicals		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6

4,8-dioxa-3H-perfluorononanoic acid	DONA	919005-14-4
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	F53B Major (9Cl-PF3ONS)	756426-58-1
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	F53B Minor (11Cl-PF3OUdS)	763051-58-1

*Certification not offered by PAB (NYSDOH)

The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Working Range
Water	250 mL	2.0 ng/L - 400 µg/L

2.0 Summary of Method

Samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an [redacted] solution.

The final [redacted] extracts are analyzed by LC/MS/MS operated in electrospray (ESI) negative ion mode. PFAS are separated from other components on a C18 column with a solvent gradient program using [redacted] and methanol.

An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDAs) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compound of interest, and they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have an identically labeled analog are quantified by the IDA method using a closely related labeled analog.

Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

This SOP is based on the following reference methods:

- Method ISO 25101, “Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry”, First Edition, 2009-03-01, International Organization for Standardization, Technical Committee ISO/TC 147, Water Quality, Subcommittee SC 2, Physical, chemical and biochemical methods.

If the laboratory’s SOP is modified from the reference method, a list of method modifications along with technical justification may be found in Section 16. Modifications to this SOP may be applied on a project specific basis to meet project data quality objectives. Project specific modifications are documented in the project record.

3.0 Definitions

Refer to the Laboratory's Quality Assurance Manual (QAM) for the Glossary of Terms, Definitions and Acronyms except as follows:

Definitions of terms used in this SOP may be found in Appendix A.

4.0 Interferences

PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFAS contamination.

Standards and samples are injected from polypropylene autosampler vials with polyethylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

Random evaporation losses have been observed with the polyethylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same Teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polyethylene screw caps.

Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFAS. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

Both branched and linear isomers of PFOS, PFOA, PFHxS, PFBS, EtFOSAA and MeFOSAA can potentially be found in the environment, based upon scientific literature. If multiple isomers are present for one of these PFAS, these adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting, and all the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included.

When reference standards of technical mixtures of specific PFAS area available, they should be used to ensure that all appropriate peaks are included during peak integration (at this time, only PFOS, PFOA, PFHxS, EtFOSAA and MeFOSAA are available as technical mixtures). Refer to Section 7, Reagents, for the available technical mixtures utilized by this SOP.

In an attempt to reduce PFOS bias, it is required that m/z 449>80 transition be used as the quantitation transition.

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

Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS must be handled in the laboratory as hazardous and toxic chemicals.

Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

Laboratory procedures such as the use of pipets and transferring of extracts represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

Eye protection that satisfies ANSI Z87.1 (as per the Eurofins TestAmerica Corporate Safety Manual), a laboratory coat and nitrile gloves must be worn while handling samples, standards, solvents and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

Perfluorocarboxylic acids are acids and are not compatible with strong bases.

The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

The HPLC and MS/MS have areas of high voltage. Depending on the type of work involved, the instrument should be turned off or disconnected from its source of power prior to extensive maintenance.

5.2 Primary Materials Used

Table 2 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as

identified in the SDS. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

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

6.1 Miscellaneous

- 15 mL polypropylene test tubes with screw caps, Fisherbrand 05-539-5 or equivalent.
- 250-mL HDPE wide-mouth bottles with screw caps (ESS 0250-1901-).
- Analytical balance capable of weighing to the nearest 0.01g, and checked for accuracy each day it is used in accordance with BR-GT-008.
- SPE Vacuum manifold, 24-port, [redacted] or equivalent.
- 1/8” OD Poly siphon lines, 30” long for sample loading.
- SPE Adaptor Caps for 1, 3, and 6 mL SPE Tubes, Polyethylene, [redacted], or equivalent.
- SPE Stopcocks, Polyethylene and Polypropylene, [redacted], or equivalent.
- Stainless steel solvent guide needles, [redacted], or equivalent.
- Heavy-Wall filter flask, Fisherbrand 4000mL, [redacted], or equivalent.
- Polypropylene Syringe, 10 mL with luer-lok or luer slip tips, Norm-Ject AB10LL or equivalent.
- Volumetric Syringes, Class “A” (25µL, 50µL 100µL, and 500µL), Hamilton or equivalent.
- Automatic Pipettor, Finnpette, 1-5mL.
- Polypropylene autosampler vials, 300µL, 700µL and 2mL with polyethylene screw caps.
- Waters Oasis [redacted]) or equivalent.
- Vacuum manifold for Solid Phase Extraction (SPE).
- 250mL Poly bottles containing 1.25g of Trizma Pre-Set Crystals, used for batch QC for samples received with Trizma preservation.
- 500ml Polyethylene wash bottle
- 4, 6, and 12ml Class A Volumetric Pipette
- Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc). These should be disposable wherever possible, or marked and segregated for high-level versus low-level use.

6.2 Analytical System

Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS)-as described below. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent

- [redacted] LC/MS/MS
  This system consists of a [redacted] HPLC interfaced with a [redacted] Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalvent.

  [redacted] HPLC equipped with [redacted] pumps and one [redacted] degassing unit or equivalent. [redacted] Column Oven.

  [redacted], or equivalent.

  PFAS Isolator column. Restek Ultra C18 5µm, 10 x 2.1mm, two aligned in series. These are plumbed between the pump's mixing valve and the autosampler to minimized the HPLC-based PFAS background from injection-based PFAS.

7.0 Reagents and Standards

7.1 Reagents

All reagents must follow traceability guidelines found in SOP BR-QA-002.

- Ammonium acetate Stock Solution, [redacted]
- [redacted]-[redacted].
- Ammonium hydroxide, concentrated, JT Baker or equivalent.
- Ammonium hydroxide (NH_4OH) [redacted].
- Reagent Water, house reverse-osmosis reagent water ("PFAS-Free" via in-house testing).
- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.
- Methanol, HPLC JT Baker or equivalent.
- Sodium hydroxide, pellets, JT Baker or equivalent.
- Sodium hydroxide (NaOH), [redacted]
- Acetonitrile, Optima Grade, Fisherbrand or equivalent.

7.2 Standards

Purchase high purity, technical grade solids (96% or greater) or certified solutions from commercial vendors. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at 4 ± 2°C. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

As of this writing, only PFOS, PFOA, PFHxS, MeFOSAA and EtFOSAA are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

PFBS, PFHxS, PFHpS, PFOS, PFDS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$$Mass_{acid} = \text{Measured } Mass_{salt} \times MW_{acid} / MW_{salt}$$

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be multiplied by a factor of 0.956 to account for the amount of PFOS in the final solution.

While PFAS standards commercially purchased are supplied in glass ampoules, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene or HDPE containers.

Prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

A technical (qualitative) grade PFOA standard is analyzed initially, then after initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

A second source solution for PFAS is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. For those compounds not available in this mixture or are not available from another vendor, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IDA and ISTD are added at a fixed concentration (2.5 ng/mL in extract).

7.3 Extraction Spiking Solutions

PFAS LCS/Matrix Spike Solution, 400 ng/mL

The PFAS spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 400 ng/mL in methanol.

PFAS High Level LCS Solution, 1000 ng/mL

The PFAS spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 1000 ng/mL in methanol.

PFAS Isotope Dilution Analyte Solution, 1000 ng/mL

The PFAS-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each IDA compound at a concentration of 1000 ng/mL in methanol.

Internal Standard Solution, $^{13}C_2$-PFOA, 2500 ng/mL

The internal standard solution is prepared by diluting the stock 50 µg/mL $^{13}C_2$-PFOA 20-fold in methanol.

See Appendix B for analyte lists and concentrations.

8.0 Sample Collection, Preservation, Shipment and Storage

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

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	Minimum Sample Size	Preservation	Holding Time[1]
Drinking Water	250 mL HDPE Bottle	250 mL	0-6°C Trizma (5g/L) (if from a known chlorinated source)	14 days from collection
Extract	700 µL Polypropylene (PP) Vial with HDPE Screw cap	NA	0-6°C	40 days from extraction

[1] *Extraction holding time is calculated from date of collection. Analytical holding time is determined from date of extraction.*
[2] *TestAmerica Sacramento has conducted holding time studies that support a 14 day holding time for aqueous samples. The 14 /40 day holding times given above are based on the holding time study and general EPA convention for the holding time of extractable organic compounds in water and soil.*

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

When samples contain the preservative Trizma, all associated QC must be treated with the same preservative.

Initial Demonstration of Capability (IDOC) and Method Detection Limit (MDL) studies described in Section 12 must be acceptable before analysis of samples may begin.

Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence.

The laboratory prepares the following sample QC for each extraction batch (an extraction batch is limited to a maximum of 20 field samples of the same matrix processed using the same procedure and reagents within the same time period):

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 per extraction batch	See Table 3
Laboratory Control Sample (LCS)	1 per extraction batch	See Table 3
Laboratory Control Sample Duplicate (LCSD)	1 per extraction batch whenever sufficient sample is available for an MS/MSD	See Table 3
Matrix Spike (MS/MSD)	1 per extraction batch (if sufficient sample is available)	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

Note: When samples are received with Trizma preservation, the MB, LCS and LCSD (if needed) must be prepared in reagent blank aliquots that have been Trizma preserved. It is recommended to keep Trizma and non-Trizma samples segregated in separate preparation batches.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially, when CCV fails and after major instrument maintenance	See Table 3
Initial Calibration Blank (ICB)	Immediately after ICAL	See Table 3
Second Source Verification (ICV)	Immediately after ICB	See Table 3
Continuing Calibration Verification (CCV)	Beginning, end and after every 10 field samples. Alternate between ICAL Levels 4, and 5 (in order) throughout sequence	See Table 3
Continuing Calibration Verification Low (CCVL)	Immediately prior to Level 4 CCV at beginning of every non-ICAL analytical sequence	See Table 3
Isotope Dilution Analytes (IDA)	Added to Every injection (Standards, QC and Field Samples) at the same concentration	See Table 3

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or

other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP BR-QA-0016. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

If samples are not collected in 250 mL HDPE bottles, transfer approximately 250 mL of each sample into a new, labeled 16 oz. polyethylene (HDPE) bottle and write an NCM in the preparation batch indicating that the sample was collected in the wrong container, and the container walls could not be extracted. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS. NOTE: If any of the samples in a job have been collected in Trizma preserved bottles, separate batch QC (MB, LCS and LCSD, if necessary) must be prepared using Trizma preserved bottles in a separate prep batch.

Weigh each container to determine its pre-extraction mass (Gross Weight). Record this value directly into the TALS batch.

Spike the LCS and MS/MSD (if requested) with 0.025 mL (25 µL) of the PFAS LCS/Matrix Spike solution (Section 7.2). This will result in a sample concentration of 40 ng/L.

Add [redacted] of the PFAS-IDA solution (Section 7.2) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

Recap the bottles and shake to mix the contents. After the extraction has been completed, allow the container to completely dry (uncapped). Replace the cap and reweigh the container to determine the container mass (Tare Weight). The sample volume extracted can be determined by subtracting the Tare Weight from the Gross Weight. These calculations are captured in the PFAS water sample prep module (25101_2009_SPE).

Due to the surface active nature of the PFAS analytes, it is necessary to extract the entire sample as well as the container walls to maximize recovery. It is therefore ideal to receive full 250 mL HDPE bottles for each sample (and MS/MSD when requested) so the entire sample can be processed from that container.

If the sample is received with sediment, it may not be possible to extract the entire sample. In this case the laboratory will spike the entire volume received and will attempt to extract at least 50mL of sample. If the SPE cartridge clogs prior to the entire sample eluting through, the container walls will not be extracted. The reduced extraction volume will be noted with an NCM.

10.2 Solid Phase Extraction (SPE)

Condition the SPE cartridges [redacted] by passing the following without drying the column.

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

Wash with 5.0 mL of [redacted].

Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 1 mL remains on top to keep column wet. After this step, the columns should not go dry until the completion of loading and rinsing samples.

Appropriately label the SPE cartridges.

Add a poly siphon line to an adapter which has been firmly inserted into the SPE cartridge and place the other end of the line into the corresponding sample container.

Turn on the vacuum and pull the entire sample volume (minimum of 250 mL) through the cartridge at rate of approximately [redacted].

Stop the sample elution when ~0.1 mL remains. Add ~5 mL of water to the SPE column and restart the elution to complete the loading process. The added water volume ensures there are no small sample droplets remaining that may be clinging to the wall of the SPE cartridge.

After the sample and water rinse has passed through the cartridge, allow the cartridge to completely dry with vacuum (this could take up to 90 minutes). The cartridge should return to a uniform color. NOTE: Remove and replace each cartridge during the drying process to ensure any water droplets that may be in the flow path are eliminated.

10.3 SPE Column Wash of with Hexane

Add [redacted] to each SPE column and let the column become fully saturated with solvent. Close the stopcock and allow the column to soak for five minutes, then elute to waste.

Load a second [redacted] and elute to waste (without a soaking period).

Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried thoroughly before continuing. The cartridge should return to a uniform color. Wipe any remaining water droplets from the bottom of the stainless steel guide needles using a fresh Kimwipe for each needle prior to proceeding to the next step.

10.4 SPE Elution

Note: the use of glass should be avoided where able. However, disposable glass pipettes have a much narrower opening, which is necessary to reduce spillage during the following transfer steps.

Place labeled 15 mL polypropylene test tubes containing [redacted] of Reagent Water as receiving tubes in the SPE manifold.

Rinse the dried sample bottles with [redacted] and transfer to the corresponding SPE cartridge using a disposable glass pipet (NOTE: the sample container has molded ridges in the neck that can trap up to 0.5mL of the solvent rinsate; make sure to tip the container slightly to draw the rinsate out of the ridges). Allow the solution to soak the cartridge for

5 minutes and then elute into the 15 mL collection tube.

Repeat the sample bottle rinse to cartridge elution process with a [redacted] (without the soaking period) The total collection should be approximately 10 mL. Adjust to 10 mL with methanol.

10.5 Internal Standard Addition

Add [redacted] internal standard to each extract and vortex to mix well.

Transfer a portion of the extract to a labeled 300µL polypropylene autosampler vial (6 drops or approximately 60µL). Archive the rest of the extract in the event the sample needs re-injection and/or dilution.

Seal the vials with polyethylene screw caps. Note: Teflon lined caps may not be used due to detection of low level concentration of PFAS.

10.6 Instrument Operating Conditions

Suggested operating conditions are listed below for the [redacted] LCMS system:

Recommended Instrument Operating Conditions					
HPLC Conditions ([redacted] HPLC)					
Column (Column temp = 45°C)	Phenomenex Gemini C18 3um, 3.0mm x 100mm				
Mobile Phase Composition	A=20mM Ammonium Acetate (90/10 water/methanol) B=Methanol				
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate mL/min.**
	[redacted]			6	0.60
				6	0.60
				6	0.60
				6	0.60
				6	0.60
				6	0.60
	Maximum pressure limit = 5,000 psi				
Injection Size	[redacted]				
Run Time	[redacted]				
Mass Spectrometer Interface Settings [redacted]					
MS Interface Mode	[redacted]				
Ionspray (volts)	[redacted]				
Declustering Potential-DP (volts)	[redacted]				
Entrance Potential-EP (volts)	[redacted]				
Source Temp (TEM)	[redacted]				
Curtain Gas (CUR)	[redacted]				
Collision Gas (CAD)	[redacted]				
Ion Source Gas 1 (GS1)	[redacted]				
Ion Source Gas 2 (GS2)	[redacted]				
Collision Energy-CE (volts)	[redacted]				

Collision Cell Exit Potential-CXP (volts)	[redacted]

Recommended Instrument Operating Conditions

Mass Spectrometer Scan Settings (SCIEX 5500 QQQ)

Compound	Comments	Reaction (MRM)	Dwell (sec)	DP(v)	EP(v)	CE(v)	CXP(v)
PFBA	Native analyte	212.9 > 169.0	0.011	[redacted]			
13C4 PFBA	IDA	217.0 > 172.0	0.011				
PFPeA	Native analyte	262.9 > 219.0	0.011				
13C5 PFPeA	IDA	267.9 > 223.0	0.011				
PFBS	Native analyte	298.9 > 80.0	0.011				
PFBS_2	Native analyte	298.9 > 99.0	0.011				
13C3 PFBS	IDA	301.9 > 80.0	0.011				
PFHxA	Native analyte	313.0 > 269.0	0.011				
PFHxA_2	Native analyte	313.0 > 119.0	0.011				
13C2 PFHxA	IDA	315.0 > 270.0	0.011				
4:2FTS	Native analyte	327.0 > 307.0	0.011				
M2-4:2FTS	IDA	329.0 > 81.0	0.011				
PFPeS	Native analyte	349.0 > 80.0	0.011				
PFPeS_2	Native analyte	349 > 99.0	0.011				
HFPO-DA	Native analyte	329.1 > 285	0.011				
13C3 HFPO-DA	IDA	332.1 > 287	0.011				
PFHpA	Native analyte	363.0 > 319.0	0.011				
PFHpA_2	Native analyte	363.0 > 169.0	0.011				
13C4 PFHpA	IDA	367.0 > 322.0	0.011				
PFHxS	Native analyte	399.0 > 80.0	0.011				
PFHxS_2	Native analyte	399.0 > 99.0	0.011				
18O2 PFHxS	IDA	403.0 > 84.0	0.011				
DONA	Native analyte	377 > 251	0.011				
DONA_2	Native analyte	377 > 85	0.011				
PFOA	Native analyte	413.0 > 369.0	0.011				
PFOA_2	Native analyte	413.0 > 169.0	0.011				
13C2 PFOA	Internal Std	415.0 > 370.0	0.011				
13C4 PFOA	IDA	417.0 > 372.0	0.011				
6:2FTS	Native analyte	427.0 > 407.0	0.011				
M2-6:2FTS	IDA	429.0 > 81.0	0.011				
PFHpS	Native analyte	449.0 > 80.0	0.011				
PFHpS_2	Native analyte	449.0 > 99.0	0.011				
PFNA	Native analyte	463.0 > 419.0	0.011				
PFNA_2	Native analyte	463.0 > 169.0	0.011				
13C5 PFNA	IDA	468.0 > 423.0	0.011				
PFOS	Native analyte	499.0 > 80.0	0.011				
PFOS_2	Native analyte	499.0 > 99.0	0.011				
9Cl-PF3ONS	Native analyte	531 > 351	0.011				

13C4 PFOS	IDA	503.0 > 80.0	0.011	
PFDA	Native analyte	513.0 > 469.0	0.011	
PFDA_2	Native analyte	513.0 > 169.0	0.011	
13C2 PFDA	IDA	515.0 > 470.0	0.011	
8:2FTS	Native analyte	527.0 > 507.0	0.011	
M2-8:2FTS	IDA	529.0 > 81.0	0.011	
PFNS	Native analyte	549.0 > 80.0	0.011	
PFNS_2	Native analyte	549.0 > 99.0	0.011	
MeFOSAA	Native analyte	570 > 419.0	0.011	
d3-MeFOSAA	IDA	573.0 > 419.0	0.011	
11Cl-PF3OUdS	Native analyte	631 > 451	0.011	
FOSA	Native analyte	498.0 > 78.0	0.011	
13C8 FOSA	IDA	506.0 > 78.0	0.011	
PFUdA	Native analyte	563.0 > 519.0	0.011	
PFUdA_2	Native analyte	563.0 > 169.0	0.011	
13C2 PFUdA	IDA	565.0 > 520.0	0.011	
EtFOSAA	Native analyte	584.0 > 419.0	0.011	
d5-EtFOSAA	IDA	589.0 > 419.0	0.011	
PFDS	Native analyte	599.0 > 80.0	0.011	
PFDS_2	Native analyte	599.0 > 99.0	0.011	
PFDoA	Native analyte	613.0 > 569.0	0.011	
PFDoA_2	Native analyte	613.0 > 169.0	0.011	
13C2 PFDoA	IDA	615.0 > 570.0	0.011	
PFTrDA	Native analyte	663.0 > 619.0	0.011	
PFTrDA_2	Native analyte	663.0 > 169.0	0.011	
PFTeDA	Native analyte	713.0 > 669.0	0.011	
PFTeDA_2	Native analyte	713.0 > 169.0	0.011	
13C2 PFTeDA	IDA	715.0 > 670.0	0.011	

Recommended Instrument Operating Conditions				
Retention Times & Quantitation [redacted]				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	[redacted]	13C4 PFBA	[redacted]	Isotope Dilution
PFPeA		13C5 PFPeA		Isotope Dilution
PFBS		13C3 PFBS		Isotope Dilution
4:2FTS		M2-4:2FTS		Internal Standard
PFHxA		13C2 PFHxA		Isotope Dilution
PFPeS		13C3 PFBS		Internal Standard
HFPO-DA		13C3 HFPO-DA		Isotope Dilution
PFHpA		13C4 PFHpA		Isotope Dilution
PFHxS		18O2 PFHxS		Isotope Dilution
DONA		13C4 PFOS		Internal Standard
6:2FTS		M2-6:2FTS		Isotope Dilution
PFOA		13C4 PFOA		Isotope Dilution
PFHpS		13C4 PFOS		Internal Standard
PFNA		13C5 PFNA		Isotope Dilution
PFOS		13C4 PFOS		Isotope Dilution
9Cl-PF3ONS		13C4 PFOS		Internal Standard
8:2FTS		M2-8:2FTS		Isotope Dilution
PFDA		13C2 PFDA		Isotope Dilution
PFNS		13C4 PFOS		Internal Standard
MeFOSAA		d3-MeFOSAA		Isotope Dilution
11Cl-PF3OUdS		13C4 PFOS		Internal Standard
EtFOSAA		d5-EtFOSAA		Isotope Dilution
PFDS		13C4 PFOS		Internal Standard
PFUdA		13C2 PFUdA		Isotope Dilution
FOSA		13C8 FOSA		Isotope Dilution
PFDoA		13C2 PFDoA		Isotope Dilution
PFTrDA		13C2 PFTeDA		Internal Standard
PFTeDA		13C2 PFTeDA		Isotope Dilution

Note: clients must be notified when the quantitation of an analyte is performed using an Internal standard. Changes to these IDA/ISTD associations may be necessary when sources of IDAs are updated: this may include additions as new IDAs become available, or subtractions if IDAs are unavailable.

10.7 Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native and IDA) into the MS/MS electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table above.

10.8 Instrument Calibration

Perform initial calibration with a minimum of five calibration standards before any sample analysis (initial method set-up), whenever a new column is installed, when significant instrument maintenance has been performed, and when the CCV does not meet acceptance criteria. Significant instrument maintenance includes installing a new column, changing the proportioning valve, or changing components of the MS/MS system. A new calibration is not required following minor maintenance.

With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points. The same injection volume must be used for all injections (standards and extracts).

Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by isotope dilution must be < 20% for the curve to be valid.

For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by internal standard (i.e. those compounds that do not have corresponding isotopically labeled analogs) must be < 25% for the curve to be valid.

For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

-Response must increase with increasing concentration.

-The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.

-There should be no carryover at or above 1/2 MRL after a high CAL standard.

-The low cal. point must recover to within 50-150%, and all others must recover to within 70-130%.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.8.1 Initial Calibration

Prepare the working calibration standards using the recommended formulations given in Appendix B ensuring the lowest calibration standard for each analyte is equal to or below the established RL. Unless otherwise specified on a project basis, use calibration levels 1 to 6 to establish the calibration curve for each analyte.

Prime the instrument by analyzing a minimum of 4 “primer” solutions consisting of 80/20 methanol/water. In general, an HPLC contains components made from PTFE, which enable the pumps to work with many types of organic solvents. Despite efforts to remove as much PTFE as possible, certain components cannot be replaced and contribute PFAS. The longer the system remains idle, the more PFAS that is yielded. Therefore these primers serve to reduce and stabilize the amount of PFAS that are contributed. Immediately following the primers is a Blank, the ICAL sequence (run in ascending order of Level 1 to Level 6), the ICB, the ICV and the first analytical window of extracts (up to 10 field samples). The data is acquired using using Sciex’s Analyst 1.6.

The Chrom Review data system generates calibration data by generating relative response factors (RRFs) based on the response of the target analyte and its corresponding Isotope Dilution Analyte (or Internal Standard) as well as their injection concentrations to ultimately generate Mean Response Factors. PFOA and PFOS calibrated using IDA must have RSD values <20% (<35 for all other IDA calibrated analytes), Compounds calibrated using ISTD must have RSD values < 50%. The IDA compounds are also calibrated using an internal standard.. The IDA RSD for ^{13}C labeled PFOA and PFOS must be <20% (the RSD for all other labeled PFAS must be < 50%). Alternatively, a linear regression curve of concentration vs. peak area for each analyte relative to their corresponding IDA/ISTD and their concentrations calculates the correlation coefficient with 1/concentration weighting. The calibration must have a correlation coefficient (r) $\geq$ 0.995 ($r^2 \geq 0.990$). If criteria are not met, correct the problem and repeat calibration. Further analysis may not proceed without valid calibration.

10.8.2 Initial Calibration Blank (ICB)

Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of [redacted]ortified with IDA solution and ISTD solution at 50 ng/mL

The result for the calibration blank must be less than the reporting limit.

If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.8.3 Second Source Calibration Verification (ICV)

Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

The recovery for the ICV must meet the appropriate following criteria:

The native analyte must be within or equal to 70-130% for all native analytes quantitated by isotope dilution.

The native analyte must be within or equal to 70-130% for all native analytes quantitated by internal standard (i.e. those compounds that do not have corresponding isotopically labeled analogs).

The IDA recovery must be within or equal to 50-150%.

See Table 3 for corrective actions in the event that the ICV does not meet the criteria above.

10.8.4 Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid-level range of the curve and should vary throughout the run. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition, the low standard in the curve must be analyzed and must be within ± 50% of the expected value.

The native PFOA and PFOS recovery for the CCV standards must be 80-120% (70-130% for all other natives quantitated by isotope dilution) and 60-140% for all natives quantitated by internal standard. The recovery for ^{13}C labeled PFOA and PFOS must be 80-120% (the other IDA must be within or equal to 50-150%).

If this is not achieved, the instrument has drifted outside the calibration limits. If the CCV fails again following minor maintenance, the instrument must be recalibrated.

10.8.5 Isotope Dilution Analytes (IDA)

The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 10.1. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

IDA recoveries are flagged if they are outside of the acceptance limits (70-130% for ^{13}C PFOA/PFOS and 25–150% for all other labeled PFAS). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.

Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

10.9 Troubleshooting:

Check the following items in case of calibration failures:

Evaluate the failure to determine whether it affects all of the compounds in the ICAL equally. If one ICAL point appears low or high, re-prep the curve and rerun, as the error was most likely prep-based. If only a subset of the analytes are affected, check the integration and chromatography to see if there are anomalies; if justifiable, correct the integration so it is consistent with the other ICAL levels.

If there are no peaks for all compounds or no peaks after a specific retention time, ensure that the HPLC pump is pumping properly; it may have shut down due to overpressure or has a leak. If the pump has shut down, confirm it is primed and replace the in-line filter. If the pressure climbs above expected levels, changing the guard column and even analytical column may be necessary. It's best to chase high pressure sources from the pump forward (ie the post-pump in-line filter, isolator column, post-autosampler in-line filter, guard column, analytical column and MSMS inlet. If the pump is still pumping, check the system pressure. If it is lower than expected, check for leaks. Start with all connections, then move on to pump seals, especially if there are wide variations in pressure when pumping the same solvents at the same flow rates. If the pump is still pumping and the pressure is normal, check to make sure the MSMS is still functioning properly. Most issues with the MSMS system will be noted by the instrument software.

If there are peaks for all analytes, evaluate the peak shapes by comparing them to the ICAL chromatography. If the peaks have changed (shorter and wider), a new guard column may improve peak shape and bring the system back into compliance. If a new column is necessary, a new ICAL will be needed.

Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance
As Needed: Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.
Daily (When in use) Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that multiplier is functioning.

10.10 Sample Analysis

Place the field and QC samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

An example analytical sequence that includes initial calibration (ICAL) is provided below.

Injection Number	Lab Description
1	Primer 1
2	Primer 2
3	Primer 3
4	Primer 4
5	Blank
6	Calibration Level 1
7	Calibration Level 2
8	Calibration Level 3
9	Calibration Level 4 (ICIS)
10	Calibration Level 5
11	Calibration Level 6
12	ICB
13	ICV
14	T-PFOA
15	MB
16	LCS
17-26	(up to) 10 Field samples
27	CCV L4
28-37	(up to) 10 Field samples
38	MS
39	MSD
40	CCV L5
41	MB
42	LCS
43-52	(up to) 10 Field samples
53	CCV L4
54-63	(up to) 10 Field samples
65	MS
66	MSD
67	CCV L5

An example analytical sequence without ICAL:

Injection Number	Lab Description
1	Primer 1
2	Primer 2
3	Primer 3
4	Primer 4

5	CCB
6	CCVL (LOQV)
7	CCVIS (L4)
8	MB
9	LCS
10-19	(up to) 10 Field samples
20	CCV L5
21-30	(up to) 10 Field samples
31	MS
32	MSD
33	CCV L4
34	MB
35	LCS
36-45	(up to) 10 Field samples
46	CCV L5
47-56	(up to) 10 Field samples
57	MS
58	MSD
59	CCV L4

Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and initiate the analytical sequence.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards. The retention times of PFAS with labeled standards must be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards. *Note: The IS RT and native RT may be offset by 0.02 to 0.04 minutes.*

Quantitative Identification

The ICAL established in Section 10.7 is used to calculate concentrations for the extracts. The data processing system determines on-column concentration. Final results are calculated by the laboratory's LIMS information system (TALS).

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Check the results of samples analyzed immediately after high concentration samples (those with results above calibration range) for signs of carry-over. Reanalyze all samples suspected of carry -over.

12.2 Calculations

See Appendix C.

12.3 Data Review

Refer to laboratory SOP BR-QA-019 for additional instruction on the requirements for data review. The following sections summarize the general procedure as described in the data review SOP.

12.3.1 Primary Review

Review the chromatography and quantitation in the data processing system to confirm quantitative and qualitative identification of each target analyte. Perform and document manual integrations only if needed per the instructions in corporate policy CA-Q-S-002, Acceptable Manual Integration Practices.

Upload the data files to TALS and process the batch. Enter job information into the batch editor and add the standards and reagent additions to the worksheet, if necessary. Review the results against acceptance criteria. If acceptance criteria are not met, perform corrective action or make arrangements for corrective action with another analyst.

Set results to primary, secondary, acceptable or rejected. Set results to be reported to a status of primary and secondary. Set results that meet criteria but will not be reported to acceptable. Set results that do not meet criteria to rejected, to prevent inadvertent reporting of data.

Verify that all appropriate QC were performed and acceptable. If insufficient volume is received (MS, MSD, FRB, etc…) document in an NCM. Record all instances where acceptance criteria are not met in a nonconformance memo (NCM).

Verify that all project requirements or program specific requirements were followed. If not, immediately notify the project manager to determine an appropriate course of action. Record decisions made in the data review checklist.

Set the batch to 1st level review. Complete the data review checklist and make arrangements for secondary review by a peer analyst.

12.3.2 Secondary Data Review (Performed by Peer Analyst)

Record review using the data review checklist.

Verify that all project requirements or program specific requirements were followed. If not, consult with the primary analyst to determine cause. Any decisions made should be recorded on the data review checklist and retained as part of the analytical record.

Review the TALS batch editor to verify ancillary information for the work performed is filled in.

Verify that that the procedures in this SOP were followed. If discrepancy between the SOP and the analytical record is found, consult with the primary analyst to determine the source of the discrepancy. Resolve the discrepancy and verify any modifications to the SOP are properly documented and were approved by laboratory management. Record all SOP deviations in an NCM.

Spot-check ~15% of samples in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in SOP CA-Q-S-002.
- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in SOP CA-Q-S-002.
- If an error is suspected, the reviewer must consult with the analyst that performed the integration to determine if a correction is necessary. Input from the Technical Manager (TM), Department Manager (DM), or QA Manager (QAM) may be sought as necessary. **The reviewer may not reintegrate except in those circumstances approved by laboratory management**, such as when the analyst that performed the integration is on vacation. If re-integration is performed by the reviewer, the reviewer is now considered the "primary analyst" and the re-integration is subject to the same review and documentation requirements as the original integration.

Verify acceptance criteria were met. If not, verify that corrective actions were performed and the nonconformance was documented with an NCM. Review the NCM to verify the form is filled out and the requisite information has been included in the internal comments tab. If corrective action was not performed and the failure not documented, consult with the primary analyst to determine cause. Consult with the primary analyst and department management to determine what actions should be taken, then follow-through with the decision made.

Run the QC checker and fix any problems found. Run and review the deliverable for gross error such as missing data. Fix any problems found.

When review is complete set the method chain to lab complete. Complete the data review checklist and forward associated paperwork to report/project management.

12.3.3 Data Reporting & Record Retention

The specifications for data reporting are set by the project manager and are performed by TALS using the formatter selected by the PM. The type of deliverable is also set by the PM based on various deliverable options in the TALS system. The formatters and deliverables are programmed into TALS by corporate IT staff and cannot be modified locally.

The following sections describe the default reporting scheme set for this method in TALS:

Data is retained, managed and archived as specified in laboratory SOP BR-QA-014 Laboratory Records.

13.0 Method Performance

13.1 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. An initial method detection limit study is performed in accordance with SOP BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample can be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP BR-QA-011.

14.0 Pollution Control

It is Test America's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.0 Waste Management

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

- Vials containing sample extracts: Satellite Container: 30 gallon poly barrel located under GC-Semi prep hood.
- Solvent Waste: Satellite Container: 5 gallon poly carboy located under LCMSMS.

16.0 References / Cross References

- TestAmerica Sacramento SOP WS-DW-0005r1.1, "Per- and Polyfluorinated Substances (PFAS) in Potable Water [Method ISO 25101:2009]".
- Method ISO 25101, "Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry", First Edition, 2009-03-01, International Organization for Standardization, Technical Committee ISO/TC 147, Water Quality, Subcommittee SC 2, Physical, chemical and biochemical methods.
- Laboratory SOP BR-QA-005 *Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).*
- Laboratory SOP BR-QA-011 *Employee Training*
- Laboratory SOP BR-EH-001 *Hazardous Waste*
- Laboratory SOP BR-QA-014 *Laboratory Records*
- Corporate SOP CA-Q-S-002 *Manual Integration*
- Laboratory Quality Assurance Manual (QAM)

17.0 Method Modifications

Modification Number	Method Reference	Modification & Technical Justification
1	Section 7.2	Method 25101 specifies that the values reported for PFOA and PFOS shall be the linear isomer only. In keeping with the dictates of USEPA 537 and other US conventions, the laboratory reports both the branched (when present) and linear isomers as a single value for these compounds.
2	Section 10.1	Method 25101 specifies that 500mL of sample be extracted. The laboratory extracts 250mL of sample and has demonstrated the ability to produce the desired 2 ng/L reporting limit for PFOA and PFOS
3	Section 10.1	A different SPE cartridge, Waters OASIS WAX, is used for the extraction process. As a result, solvents and elution procedures are different.
4	Section 10.5	The HPLC Column, Eluents and gradient conditions have changed.
5	Section 10.5	The analyte list has expanded. The number of labeled analytes has also expanded to improve quantitation.

6	Section 10.7.1	The acceptance criteria for both the initial and continuing Calibration has changed due to the use of IDA and external standard quantitation.
7	Table 1	The reporting limits have changed to a consistent value.
8	Appendix B	Calibration levels have been changed so all levels have the same analyte concentration.

18.0 Attachments

- Table 1: Routine Compound List and LOQ
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Table 4: Control Limits
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

19.0 Revision History

Rev 3.0

- Updated title page, signatures, and dates
- Section 1.1: Changed Fluorotelomer sulfonates (FTS) to acid forms.
- Section 11.0: Added corrective action requirement as it is a corporate requirement to include.
- Extended analyte list to 28 native compounds and 20 IDAs.
- Updated instrument profiles and operating procedures to include the Sciex 5500
- Updated spiking levels and calibration solutions in alignment with the Sciex 5500 capabilities which eliminates the need for extract concentration
- Removed all references to Waters instrumentation and extract concentration

Rev 2.0

- Extended analyte list to 21 native compounds and 18 IDAs.
- Altered concentration step in extract preparation by employing a reagent water keeper instead of concentrating to dryness.
- Incorporated use of internal standard for IDA recovery calculation.

Rev 1.0

- Updated title page, signatures, and dates
- Throughout: added analytes.
- Throughout: added note that TA Burlington is not certified for the additional analytes.
- Added statement of incomplete volume extraction procedure

Previous revisions are retained by the QA department.

Table 1: Routine Compound List & Limit of Quantitation (LOQ)

Compound Name	Abbreviation	CAS #	Water (ng/L)
Perfluoroalkylcarboxylic acids (PFCAs)			
Perfluoro-n-butanoic acid	PFBA	375-22-4	2.0
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3	2.0
Perfluoro-n-hexanoic acid	PFHxA	307-24-4	2.0
Perfluoro-n-heptanoic acid	PFHpA	375-85-9	2.0
Perfluoro-n-octanoic acid	PFOA	335-67-1	2.0
Perfluoro-n-nonanoic acid	PFNA	375-95-1	2.0
Perfluoro-n-decanoic acid	PFDA	335-76-2	2.0
Perfluoro-n-undecanoic acid	PFUdA	2058-94-8	2.0
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1	2.0
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8	2.0
Perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7	2.0
Perfluorinated sulfonic acids (PFSAs)			
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5	2.0
Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-4	2.0
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4	2.0
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8	2.0
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1	2.0
Perfluoro-1-nonanesulfonic acid	PFNS	68259-12-1	2.0
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3	2.0
Perfluorinated sulfonamides (FOSA)			
Perfluoro-1-octanesulfonamide	FOSA	754-91-6	2.0
Perfluorinated sulfonamidoacetic acids (FOSAA)			
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6	20.0
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9	20.0
Fluorotelomer sulfonates (FTS)			
1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*	4:2 FTS	757124-72-4	20.0
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2	20.0
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4	20.0
Fluorinated Replacement Chemicals			
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	4.0
4,8-dioxa-3H-perfluorononanoic acid	DONA	919005-14-4	2.0
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	F53B Major (9Cl-PF3ONS)	756426-58-1	2.0
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	F53B Minor (11Cl-PF3OUdS)	763051-58-1	2.0

NOTE: The LOQ values may vary. The Water LOQ is based on a 250mL nominal sample volume.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide (3-0-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. 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 damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol (2-3-0)	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.

Table 3: QC Summary, Acceptance Criteria and Recommended Corrective Action

QC Check	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action
6-Point Calibration (5 point minimum for CF and Linear Regression) (ICAL)	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF = RSD $\leq$ 20% (IDA compounds) CF = RSD $\leq$ 25% (ISTD compounds) CF = RSD $\leq$ 50% (ESTD IDA standards) Linear Regression: r $\geq$ 0.995	Correct problem and repeat initial calibration.
IDA Response	Every injection contains the IDA analytes	Field samples: 70-130% PFOA/PFOS (Other IDA 25-150%) of ICAL RF (reportable if >10x S/N ratio and >10% ICAL RF)	Standard failures must be investigated to determine the cause of the failure. Recalibration may be required. Samples with recoveries outside acceptance limits must be evaluated for data usability. Re-extraction may be necessary if data quality has been adversely affected.
IS Response	Every injection contains the IS analyte	ICAL Standards: Area of individual points must not deviate by more than 50% of ICAL mean area response Samples following ICAL: 50-150% of ICAL mean response Ongoing CCV: 50-150% of ICAL mean response Post-CCV Samples: Area must be within 50-150% of most recent CCV	Standard failures must be investigated to determine the cause of the failure. Recalibration may be required. Sample failures may be matrix related and should be evaluated to determine if the data quality has been adversely affected.
Initial Calibration Blank (ICB)	Immediately following the ICAL	< RL for all target analytes	Determine source of interference/contamination, eliminate it and recalibrate.
Second Source Standard Verification (ICV)	Prior to the analysis of samples. Generally immediately after the ICB.	CF = 70-130% (IDA targets) CF = 50-150% (ISTD targets)	Correct problem and verify second source standard. If that fails, repeat calibration.
Continuing Calibration Verification (CCV)	Beginning of each analytical sequence, every ten field samples and at the end of each analytical sequence. Alternate between levels 3, 4 and 5.	CF = 70-130% (IDA targets) CF = 60-140% (ISTD targets) ^{13}C PFOA/PFOS 80-120% Other IDA 50-150%	Rerun any samples analyzed before and after the failing CCV. Take corrective action; if subsequent CCV analyses fail, recalibrate instrument.
Continuing Calibration Verification-Low (CCVL)	Beginning of each analytical sequence that is not preceded by an ICAL to show LOQ is still valid.	CF = 50-150% (IDA targets) CF = 50-150% (ISTD targets) IDA 50-150%	Stop sample acquisition. Take corrective action; if subsequent CCV analyses fail, recalibrate instrument.
Method Blank	One per extraction batch of 20 or fewer samples	Routine: < RL for all target analytes	Reprocess MB and associated samples if any target analyte in the MB is at or above the RL, greater than 1/10 the amount detected in any sample or 1/10 the regulatory limit, whichever is greater. If the target is not greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with appropriate qualifiers. If insufficient sample is available to reprocess, report data with appropriate qualifiers.
Laboratory Control Sample	One per extraction batch of 20 or fewer samples	%R within control limits. See Table 4	Reprep and reanalyze samples for failed analytes. If reanalysis is not possible due to insufficient

QC Check	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action
			sample volume, report data with appropriate data qualifiers.
Matrix Spike / Matrix Spike Duplicate	One set per extraction batch when sufficient sample volume is provided or as requested per client	%R within control limits. See Table 4	Evaluate to determine if there is a matrix effect or analytical error. If analytical error, reanalyze or reprocess as appropriate.
Sample Duplicate	Per Client Request	RPD within control limits. See Table 4	Evaluate data to determine source for error. If analytical error is suspected, reanalyze or reprocess as appropriate.

Table 4: In-House LCS and MS/MSD Control Limits*

Analyte	In House Limits %R Water	RPD
Perfluorobutanoic acid (PFBA)	70-130	30
Perfluoropentanoic acid (PFPeA)	70-130	30
Perfluorobutanesulfonic acid (PFBS)	70-130	30
Perfluorohexanoic acid (PFHxA)	70-130	30
Perfluoropentanesulfonic acid (PFPeS)	70-130	30
Perfluoroheptanoic acid (PFHpA)	70-130	30
Perfluorohexanesulfonic acid (PFHxS)	70-130	30
Perfluorooctanoic acid (PFOA)	70-130	30
Perfluoroheptanesulfonic acid (PFHpS)	70-130	30
Perfluorononanoic acid (PFNA)	70-130	30
Perfluorooctanesulfonic acid (PFOS)	70-130	30
Perfluorodecanoic acid (PFDA)	70-130	30
Perfluorononanesulfonic acid (PFNS)	70-130	30
Perfluoroundecanoic acid (PFUdA)	70-130	30
Perfluorodecanesulfonic acid (PFDS)	70-130	30
Perfluorooctanesulfonamide (FOSA)	70-130	30
Perfluorododecanoic acid (PFDoA)	70-130	30
Perfluorotridecanoic acid (PFTrDA)	70-130	30
Perfluorotetradecanoic acid (PFTeDA)	70-130	30
Sodium 1H,1H,2H,2H Perfluorooctanesulfonate (4:2FTS)	70-130	30
Sodium 1H,1H,2H,2H Perfluorooctanesulfonate (6:2FTS)	70-130	30
Sodium 1H,1H,2H,2H Perfluorodecanesulfonate (8:2FTS)	70-130	30
N-Methyl Perfluorooctane sulfonamidoacetic acid (N-MeFOSAA)	70-130	30
N-Ethyl Perfluorooctane sulfonamidoacetic acid (N-EtFOSAA)	70-130	30
Hexafluoropropylene oxide dimer acid	70-130	30
4,8-dioxa-3H-perfluorononanoic acid	70-130	30
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	70-130	30
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	70-130	30

**The limits in this table are those in effect as of the published date of this SOP. These are default limits that will be updated once enough data has been acquired to produce more representative limits. Current in-house limits are populated in the LIMS database. Contact a laboratory representative for the most current set of limits.*

Appendix A: Terms and Definitions

PFCAs: Perfluorocarboxylic acids
PFSAs: Perfluorinated sulfonic acids
FOSA: Perfluorinated sulfonamide
PFOA: Perfluorooctanoic acid

PFOS: Perfluorooctane sulfonate
PTFE: Polytetrafluoroethylene (e.g., Teflon®)
SPE: Solid phase extraction**.**
PP: Polypropylene
PE: Polyethylene
HDPE: High density polyethylene
AFFF: Aqueous Film Forming Foam
IDA: Isotope dilution analytes
Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).
Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.
Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.
Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.
Calibration Standard: A substance or reference used to calibrate an instrument.
Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.
Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.
Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)
Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.
Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.
Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.
Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.
Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.
Matrix Spike Duplicate (MSD): a second replicate matrix spike
Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.
Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.
Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.
Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.
Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.
Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.
Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.
Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in acetonitrile using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance. For stock standards solutions made from neat material assign an expiration date of 2 years from the date of formulation.

Stock Standard Solutions

PFAS LCS/Matrix Spike Solution 1000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL) and Final Conc (ng/mL)	
PFBA	Wellington Laboratories Code: PFBA	Perfluorobutanoic acid	50	200		1000
PFPeA	Wellington Laboratories Code: PFPeA	Perfluoropentanoic acid	50	200		1000
PFBS	Wellington Laboratories Code: L-PFBS	Perfluorobutanesulfonic acid	44.2	200		884
PFHxA	Wellington Laboratories Code: PFHxA	Perfluorohexanoic acid	50	200		1000
PFPeS	Wellington Laboratories Code: L-PFPeS	Perfluoropentanesulfonic acid	46.9	200		938
PFHpA	Wellington Laboratories Code: PFHpA	Perfluoroheptanoic acid	50	200		1000
PFHxS	Wellington Laboratories Code: br-PFHxSK	Perfluorohexanesulfonic acid	45.5	200		910
PFOA	Wellington Laboratories Code: PFOA	Perfluorooctanoic acid	50	200		1000
PFHpS	Wellington Laboratories Code: L-PFHpS	Perfluoroheptanesulfonic acid	47.6	200		952
PFNA	Wellington Laboratories Code: PFNA	Perfluorononanoic acid	50	200		1000
PFOS	Wellington Laboratories Code: br-PFOSK	Perfluorooctanesulfonic acid	46.4	200		928
PFDA	Wellington Laboratories Code: PFDA	Perfluorodecanoic acid	50	200		1000
PFNS	Wellington Laboratories Code: L-PFNS	Perfluorononanesulfonic acid	48.0	200	10	960
PFUdA	Wellington Laboratories Code: PFUdA	Perfluoroundecanoic acid	50	200		1000
PFDS	Wellington Laboratories Code: L-PFDS	Perfluorodecanesulfonic acid	48.2	200		964
FOSA	Wellington Laboratories Code: FOSA-I	Perfluorooctane sulfonamide	50	200		1000
PFDoA	Wellington Laboratories Code: PFDoA	Perfluorododecanoic acid	50	200		1000
PFTrDA	Wellington Laboratories Code: PFTrDA	Perfluorotridecanoic acid	50	200		1000
PFTeDA	Wellington Laboratories Code: PFTeDA	Perfluorotetradecanoic acid	50	200		1000
4:2FTS	Wellington Laboratories Code: 4:2FTS	1H,1H,2H,2H-perfluorohexane sulfonate (4:2)	46.7	200		934
6:2FTS	Wellington Laboratories Code: 6:2FTS	1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	47.4	200		948
8:2FTS	Wellington Laboratories Code: 8:2FTS	1H,1H,2H,2H-perfluorodecane sulfonate (8:2)	47.9	200		958
NMeFOSAA	Wellington Laboratories Code: br-NMeFOSAA	N-methyl Perfluorooctane sulfonamidoacetic acid	50	200		1000
NEtFOSAA	Wellington Laboratories Code: br-NEtFOSAA	N-ethyl Perfluorooctane sulfonamidoacetic acid	50	200		1000
HFPO-DA	Wellington Laboratories Code: HFPO-DA	Hexafluoropropylene oxide dimer acid	50	200		1000
DONA	Wellington Laboratories Code: NaDONA	4,8-dioxa-3H-perfluorononanoic acid	47.1	200		942
9Cl-PF3ONS	Wellington Laboratories Code: 9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanone-1-sulfonate	46.6	200		932

11Cl-PF3OUdS	Wellington Laboratories Code: 11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonate	47.1	200		942

Solvent: Methanol

PFAS-IDA Solution (Surrogate) 1000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C4 PFBA	Wellington Laboratories Code: MPFBA	$^{13}C_4$-Perfluorobutanoic acid	50	200	10	1000
13C5-PFPeA	Wellington Laboratories Code: MPFPeA	$^{13}C_5$-Perfluoropentanoic acid	50	200		1000
13C3-PFBS	Wellington Laboratories Code: M3PFBS	$^{13}C_3$-Perfluorobutanesulfonic acid	46.5	200		930
13C2 PFHxA	Wellington Laboratories Code: MPFHxA	$^{13}C_2$-Perfluorohexanoic acid	50	200		1000
13C4 PFHpA	Wellington Laboratories Code: M4PFHpA	$^{13}C_4$-Perfluoroheptanoic acid	50	200		1000
18O2 PFHxS	Wellington Laboratories Code: MPFHxS	$^{18}O_2$-Perfluorohexanesulfonic acid	47.3	200		946
13C4 PFOA	Wellington Laboratories Code: MPFOA	$^{13}C_4$-Perfluorooctanoic acid	50.0	200		1000
13C5 PFNA	Wellington Laboratories Code: MPFNA	$^{13}C_5$-Perfluorononanoic acid	50.0	200		1000
13C4 PFOS	Wellington Laboratories Code: MPFOS	$^{13}C_4$-Perfluorooctanesulfonic acid	47.8	200		956
13C2 PFDA	Wellington Laboratories Code: MPFDA	$^{13}C_2$-Perfluorodecanoic acid	50.0	200		1000
13C8 FOSA	Wellington Laboratories Code: M8FOSA-I	$^{13}C_8$-Perfluorooctane sulfonamide	50.0	200		1000
13C2 PFUdA	Wellington Laboratories Code: MPFUdA	$^{13}C_2$-Perfluoroundecanoic acid	50.0	200		1000
13C2 PFDoA	Wellington Laboratories Code: MPFDoA	$^{13}C_2$-Perfluorododecanoic acid	50.0	200		1000
13C2 PFTeDA	Wellington Laboratories Code: MPFTeDA	$^{13}C_2$-Perfluorotetradecanoic acid	50.0	200		1000
M2-4:2FTS	Wellington Laboratories Code: M2-4:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-hexane sulfonate (4:2)	46.7	200		934
M2-6:2FTS	Wellington Laboratories Code: M2-6:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-octane sulfonate (6:2)	47.5	200		950
M2-8:2FTS	Wellington Laboratories Code: M2-8:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-decane sulfonate (8:2)	47.9	200		958
d3-NMeFOSAA	Wellington Laboratories Code: d3-M-MeFOSAA	N-methyl-d_3-perfluoro-1-octane sulfonamidoacetic acid	50.0	200		1000
d5-NEtFOSAA	Wellington Laboratories Code: d5-M-EtFOSAA	N-ethyl-d_5-perfluoro-1-octane sulfonamidoacetic acid	50.0	200		1000
M3HFPO-DA	Wellington Laboratories Code: M3HFPO-DA	$^{13}C_3$-Hexafluoropropylene oxide dimer acid	50.0	200		1000

PFAS Internal Standard Solution 5000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C2 PFOA	Wellington Laboratories Code: M2PFOA	$^{13}C_2$-Perfluorooctanoic acid	50.0	400	4	5000

Solvent: Methanol

PFAS Internal Standard Spiking Solution 2500 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C2 PFOA	Wellington Laboratories Code: M2PFOA	$^{13}C_2$-Perfluorooctanoic acid	50.0	200	4	2500

Solvent: Methanol

PFAS-IDA-IS Calibration Standards Level 1-Level 6 for the Analysis of Non-Concentrated Extracts

ICAL Level	Vol of 1ppm PFAS LCS/Matrix Spike (µL)	Vol of 100ppb Interm ICAL Solution (µL)	Nominal Conc of PFAS (ng/mL)	Vol of PFAS-IDA Solution (µL)	Conc of IDA (ng/mL)	Vol of 2.5ppm PFAS-IS SpikingSolution (µL)	Conc of IS (ng/mL)	Vol of 80/20 MeOH/H2O (µL)	Final Vol (mL)
1		2	0.050	10	2.5	4	2.5	3988	4.0
2		2	0.10	5	2.5	2	2.5	1993	2.0
3		10	0.50	5	2.5	2	2.5	1985	2.0
4		40	1.0	10	2.5	4	2.5	3950	4.0
5	10		2.5	10	2.5	4	2.5	3980	4.0
6	20		10	5	2.5	2	2.5	1975	2.0

The solvent is 80/20 Methanol/Water.

Appendix C: Equations

Initial Calibration Curve Evaluation:

The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
b = slope
c = intercept

The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

The external standard method uses the following equation:

Equation 3

$$ResponseFactor = \frac{Peak\ Area}{Concentration\ of\ Solution (ng/mL)}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 5

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

Water Sample Result Calculation:

Equation 6

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)

V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

Soil Sample Result Calculation:

Equation 7

$$\text{Concentration}, ng/g = \frac{C_{ex} V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
W_s = Weight of sample extracted (g)
D = Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \%\,\text{moisture in sample}}{100}$$ (for dry weight result)

IDA Recovery Calculation:

Equation 8

$$\%\,\text{Recovery} = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X100$$

Where ng/g = µg/kg and:

RF_{IDA} = Response Factor for IDA compound
A_t = Area response for IDA compound
A_{IS} = Area Response for IS compound
Q_{IS} = Amount of IS added
Q_t = Amount of IDA added

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(\mu g/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor =

$$\frac{SD}{\overline{CF}} \times 100\%$$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = Calculated Concentration – Theoretical Concentration x 100%
Theoretical Concentration

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$C_{extract}$ (µg/L) = $\frac{\text{Peak Area (or Height)}}{\overline{CF}}$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Solid

$$C_{sample}(ug/Kg) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\,\text{solids}} \times DF$$

Title: Per- and Poly-fluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified), PFAS by LCMSMS]

Approvals (Signature):

Don Dawicki
Laboratory Director

Kristine Dusablon
Quality Assurance Manager

Matthew Kirk
Operations Manager / EHS Coordinator

Mark Fausel
Department Supervisor

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

1.0 Scope and Application

This SOP describes the laboratory procedure for the preparation and analysis of per- and polyfluorinated substances using liquid chromatography/tandem mass spectrometry (LC/MS/MS).

Program specific requirements are not included in this SOP. The details of program specific requirements are specified in other laboratory work instructions relevant to the program.

1.1 Analytes, Matrices, and Reporting Limits

This procedure is amenable with water, soil, sediment and tissue sample matrices.
The list of target compounds that may be determined from this procedure is provided below. Table 1 presents the compounds along with their associated reporting limits (RL).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid (Perfluoro-n-butyric acid)	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA (PFUnA)	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA (PFTA)	376-06-7
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
* Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-4
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
* Perfluoro-1-nonanesulfonic acid	PFNS	68259-12-1
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
* 1H,1H,2H,2H-perfluorohexanesulfonic acid *(4:2)*	4:2 FTS	757124-72-4
1H,1H,2H,2H-perfluorooctanesulfonic acid *(6:2)*	6:2 FTS	27619-97-2

1H,1H,2H,2H-perfluorodecanesulfonic acid *(8:2)*	8:2 FTS	39108-34-4
1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2)	10:2 FTS	120226-60-0
Fluorinated Replacement Chemicals		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4,8-dioxa-3H-perfluorononanoic acid	DONA	919005-14-4
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	F53B Major (9Cl-PF3ONS)	756426-58-1
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	F53B Minor (11Cl-PF3OUdS)	763051-58-1

Abbreviations in parenthesis are the abbreviations listed in Method 537, where they differ from the abbreviation used by the laboratory's LIMS.
***Indicates the analyte is not certified in any state or program.**
Analytes with secondary certification in NJDEP can be found in Appendix D.

The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L - 20 ng/L	2.0 ng/L - 400 ng/L
Soil/Sediment	5 g	0.2 µg/Kg–2.0 µg/Kg	0.2 µg/Kg-40 µgKg
Tissue	1 g	1.0 µg/Kg–10 µg/Kg	1.0 µg/Kg–200 µg/Kg

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

Water Samples: Water samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an [redacted] solution.

Soil/sediment/tissue samples are extracted with a [redacted] solution using a TCLP tumbler operating at [redacted] The mixture is centrifuged to reduce the amount of solid transferred when decanting the solvent. The solvent extract is exchanged to water using nitrogen blowdown, then the aqueous extract is extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an ammonium hydroxide/methanol solution.

The final [redacted] extracts are analyzed by LC/MS/MS operated in electrospray (ESI) negative ion mode. PFAS are separated from other components on a C18 column with a solvent gradient program [redacted] and methanol.

An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDAs) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compound of interest, and they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have an

identically labeled analog are quantified by the IDA method using a closely related labeled analog.

Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

This SOP is based on the following reference methods:

- US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009.
- Method ISO 25101, "Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry", First Edition, 2009-03-01, International Organization for Standardization, Technical Committee ISO/TC 147, Water Quality, Subcommittee SC 2, Physical, chemical and biochemical methods.

If the laboratory's SOP is modified from the reference method, a list of method modifications along with technical justification may be found in Section 16. Modifications to this SOP may be applied on a project specific basis to meet project data quality objectives. Project specific modifications are documented in the project record.

3.0 Definitions

Refer to the Laboratory's Quality Assurance Manual (QAM) for the Glossary of Terms, Definitions and Acronyms except as follows.

Definitions of terms used in this SOP may be found in Appendix A.

4.0 Interferences

PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

Standards and samples are injected from polypropylene autosampler vials with polyethylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

Random evaporation losses have been observed with the polyethylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the

same Teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polyethylene screw caps.

Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFAS. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

Both branched and linear isomers of PFOS, PFOA, PFHxS, PFBS, EtFOSAA and MeFOSAA can potentially be found in the environment, based upon scientific literature. If multiple isomers are present for one of these PFAS, these adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting, and all the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included.

When reference standards of technical mixtures of specific PFAS area available, they should be used to ensure that all appropriate peaks are included during peak integration (at this time, only PFOS, PFOA, PFHxS, EtFOSAA and MeFOSAA are available as technical mixtures). Refer to Section 7, Reagents, for the available technical mixtures utilized by this SOP.

In an attempt to reduce PFOS bias, it is required that m/z 449>80 transition be used as the quantitation transition.

Per the Certificate of Analysis for labeled perfluorohexadecanoic acid (13C2-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. The laboratory utilizes a weighted linear regression that is not forced through the origin for the calibration of native perfluorohexadecanoic acid to account for this contribution from its labeled IDA.

5.0 Safety

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

5.1 Specific Safety Concerns or Requirements

Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS must be handled in the laboratory as hazardous and toxic chemicals.

Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

Laboratory procedures such as the use of pipets and transferring of extracts represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same

repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

Eye protection that satisfies ANSI Z87.1 (as per the Eurofins TestAmerica Corporate Safety Manual), a laboratory coat and nitrile gloves must be worn while handling samples, standards, solvents and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

Perfluorocarboxylic acids are acids and are not compatible with strong bases.

The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

The HPLC and MS/MS have areas of high voltage. Depending on the type of work involved, the instrument should be turned off or disconnected from its source of power prior to extensive maintenance.

5.2 Primary Materials Used

Table 2 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the SDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

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

6.1 Miscellaneous

- 15 mL polypropylene test tubes with screw caps, Fisherbrand 05-539-5 or equivalent.
- 250-mL HDPE wide-mouth bottles with screw caps (ESS 0250-1901-).
- Analytical balance capable of weighing to the nearest 0.01g, and checked for accuracy each day it is used in accordance with BR-GT-008.
- SPE Vacuum manifold, 24-port, [redacted] or equivalent.
- 1/8” OD Poly siphon lines, 30” long for sample loading.
- SPE Adaptor Caps for 1, 3, and 6 mL SPE Tubes, Polyethylene, [redacted], or equivalent.
- SPE Stopcocks, Polyethylene and Polypropylene, [redacted], or equivalent.
- Stainless steel solvent guide needles, [redacted], or equivalent.
- Heavy-Wall filter flask, Fisherbrand 4000mL, [redacted], or equivalent.
- TCLP tumbler, [redacted] for extraction of soil, sediment and tissue samples.
- Glass-Col ZipVap 24-port extract concentrator.

- Polypropylene Syringe, 10 mL with luer-lok or luer slip tips, [redacted] or equivalent.
- Volumetric Syringes, Class "A" (25µL, 50µL 100µL, and 500µL), Hamilton or equivalent.
- Automatic Pipettor, Finnpette, 1-5mL.
- Polypropylene autosampler vials, 300µL, 700µL and 2mL with polyethylene screw caps.
- Waters Oasis [redacted] or equivalent, for the extraction of PFAS from aqueous samples.
- Waters Oasis [redacted] or equivalent, for the cleanup of soils.
- 250mL Poly bottles containing 1.25g of Trizma Pre-Set Crystals, used for batch QC for samples received with Trizma preservation.
- 50mL graduated polypropylene centrifuge tubes. [redacted] or equivalent.
- 500ml Polyethylene wash bottle
- 4, 6, and 12ml Class A Volumetric Pipette
- Graphitized carbon (Envi-Carb™ or equivalent)
- Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc). These should be disposable wherever possible, or marked and segregated for high-level versus low-level use.

6.2 Analytical System

Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS)-as described below. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.

- SCIEX LC/MS/MS
  This system consists of a [redacted] HPLC interfaced with a [redacted]. The instrument control and data acquisition software is [redacted] or equivalvent.

  [redacted] HPLC equipped with [redacted] and [redacted] or equivalent. [redacted] Column Oven.

  [redacted], or equivalent.

  PFAS Isolator column. [redacted]. These are plumbed between the pump's mixing valve and the autosampler to minimized the HPLC-based PFAS background from injection-based PFAS.

7.0 Reagents and Standards

7.1 Reagents

All reagents must follow traceability guidelines found in SOP BR-QA-002.

- Ammonium acetate Stock Solution[redacted]
- [redacted] ammonium acetate eluent.. [redacted]

- Ammonium hydroxide, concentrated, JT Baker or equivalent.
- Ammonium hydroxide (NH_4OH) ([redacted] of Methanol. Volume prepared may be adjusted based on usage/need.
- Potassium hydroxide pellets, 87% purity, JT Baker P250-1 or equivalent.
- Potassium hydroxide (KOH), [redacted]
- Reagent Water, house reverse-osmosis reagent water ("PFAS-Free" via in-house testing).
- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.
- Methanol, HPLC JT Baker or equivalent.
- Sodium hydroxide, pellets, JT Baker or equivalent.
- Sodium hydroxide (NaOH), [redacted]
- Acetonitrile, Optima Grade, Fisherbrand or equivalent.

7.2 Standards

Purchase high purity, technical grade solids (96% or greater) or certified solutions from commercial vendors. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at 4 ± 2°C. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

Per the Certificate of Analysis for labeled perfluorohexadecanoic acid (13C2-PFHxDA) produced by Wellington Laboratories, the stock standard contains ~0.3% of native PFHxDA. This equates to roughly 0.30 ng/L or 0.015 ug/Kg of PFHxDA expected in all samples and blanks.

As of this writing, only PFOS, PFOA, PFHxS, MeFOSAA and EtFOSAA are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

PFBS, PFHxS, PFHpS, PFOS, PFDS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$$Mass_{acid} = Measured\ Mass_{salt} \times MW_{acid} / MW_{salt}$$

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be multiplied by a factor of 0.956 to account for the amount of PFOS in the final solution.

While PFAS standards commercially purchased are supplied in glass ampoules, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene or HDPE containers.

Prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The

recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

A technical (qualitative) grade PFOA standard is analyzed initially, then after initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

A second source solution for PFAS is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. For those compounds not available in this mixture or are not available from another vendor, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IDA and ISTD are added at a fixed concentration (2.5 ng/mL in extract).

7.3 Extraction Spiking Solutions

PFAS LCS/Matrix Spike Solution, 400 ng/mL

The PFAS spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 400 ng/mL in methanol.

PFAS High Level LCS Solution, 1000 ng/mL

The PFAS spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 1000 ng/mL in methanol.

PFAS Isotope Dilution Analyte Solution, 1000 ng/mL

The PFAS-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each IDA compound at a concentration of 1000 ng/mL in methanol.

Internal Standard Solution, $^{13}C_2$-PFOA, 2500 ng/mL

The internal standard solution is prepared by diluting the stock 50 µg/mL $^{13}C_2$-PFOA 20-fold in methanol.

See Appendix B for analyte lists and concentrations.

8.0 Sample Collection, Preservation, Shipment and Storage

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

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	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Water	250 mL HDPE Bottle	250 mL	0-6°C, Trizma (5g/L) (if from a known	14 days from collection	Method 537

			chlorinated source)		
Soil/Sediment	4/8 oz HDPE wide-mouth container	100 g	0-6°C	14 days from collection	SW-846 Organic Methods
Extract	700 µL Polypropylene (PP) Vial with HDPE Screw cap	NA	0-6°C	40 days from extraction	NJDEP guidance

Extraction holding time is calculated from date of collection. Analytical holding time is determined from date of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Sample QC

When samples contain the preservative Trizma, all associated QC must be treated with the same preservative.

Initial Demonstration of Capability (IDOC) and Method Detection Limit (MDL) studies described in Section 12 must be acceptable before analysis of samples may begin.

Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence.

The laboratory prepares the following sample QC for each extraction batch (an extraction batch is limited to a maximum of 20 field samples of the same matrix processed using the same procedure and reagents within the same time period):

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 per extraction batch	See Table 3
Laboratory Control Sample (LCS)	1 per extraction batch (Spiking Level rotates between Low, Medium and High on a batch-by-batch basis)	See Table 3
LCS Duplicate (LCSD)	1 per extraction batch whenever insufficient sample is available for an MS/MSD/DU	See Table 3
*Matrix Spike (MS/MSD)	1 per extraction batch (if sufficient sample is available)	See Table 3
*Sample Duplicate (SD)	DW-1 per extraction batch (if sufficient sample is available); Non-DW matrices- client request if sufficient sample is available	See Table 3
Field Reagent Blank, FRB	Per client set of samples	See Table 3

*An NCM must be applied if there is insufficient volume for a MS/MSD or duplicate.

Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially, when CCV fails and after major instrument maintenance	See Table 3
Initial Calibration Blank (ICB)	Immediately after ICAL	See Table 3
Second Source Verification (ICV)	Immediately after ICB	See Table 3
Continuing Calibration Verification (CCV)	Beginning, end and after every 10 field samples. Alternate between ICAL Levels 4 and 5 (in order) throughout sequence	See Table 3
Continuing Calibration Verification Low (CCVL)	Immediately prior to Level 4 CCV at beginning of every non-ICAL analytical sequence	See Table 3
Isotope Dilution Analytes (IDA)	Added to Every injection (Standards, QC and Field Samples) at the same concentration	See Table 3

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP BR-QA-016. The NCM shall be filed in the project file and addressed in the case narrative. ***Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.***

10.1 Water Sample Preparation

Visually inspect samples for the presence of settled and/or suspended sediment. If the amount of sediment is so great that the SPE cartridge will clog before the majority of the sample has eluted, filter the water sample through a glass fiber filter [redacted] or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank and LCS with any samples requiring filtration. File an NCM noting the need for filtration.

> Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

Due to the high surface activity of the analytes, filtration should be regarded as a last resort. All samples will be spiked with IDA prior to filtration (if enough sample is available, perform an MS on each sample); this will allow any losses caused by filtration to be monitored and corrected for.

NOTE: for samples which full volume extraction is not possible, care MUST be taken to ensure the actual sample volume that is both spiked and extracted are documented in the sample worksheet notes.

Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS.

Rotate the LCS concentration with each batch.

-Low Level LCS (50-150 %R), spike with 0.50 mL of PFAS LOQV solution. This will result in sample concentrations at the method Reporting Limit.

-Medium Level LCS (70-130 %R), spike with 0.025 mL (25 µL) of the PFAS LCS/Matrix Spike solution (Section 7.2). This will result in a sample concentration of 40 ng/L.

-High level LCS (70-130 %R), spike at 0.05mL (50uL) of the PFAS High Level LCS Spike solution (Section 7.2). This will result in a sample concentration of 200 ng/L.

Spike the MS/MSD (if available volume) with 0.025 mL (25 µL) of the PFAS LCS/Matrix Spike solution (Section 7.2). This will result in a sample concentration of 40 ng/L. NCM if there is insufficient volume to perform the MS/MSD.

Add [redacted] of the PFAS-IDA solution (Section 7.2) into each sample and QC sample, for a fixed concentration of 2.5 ng/mL in extract.

Due to the surface active nature of the PFAS analytes, it is necessary to extract the entire sample as well as the container walls to maximize recovery. It is therefore ideal to receive full 250 mL HDPE bottles for each sample (and MS/MSD if sufficient volume is received) so the entire sample can be processed from that container.

Weigh each container to determine its pre-extraction mass (Gross Weight). Spike each container in the batch with PFAS-IDA solution. Spike the LCS and LCSD (or MS/MSD, if available volume) with PFAS LCS/Matrix solution. Shake to mix the contents. After the extraction has been completed, allow the container to completely dry (uncapped). Replace the cap and reweigh the container to determine the container mass (Tare Weight). The sample volume extracted can be determined by subtracting the Tare Weight from the Gross Weight. These calculations are captured in the PFAS water sample prep module (TALS Method 3535_IVWT and 25101_2009_SPE).

10.1.1 Solid Phase Extraction (SPE) of Aqueous Samples

Condition the SPE cartridges [redacted]) by passing the following without drying the column.

> **WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.**

Wash with 5.0 mL of [redacted].

Wash with 5.0 mL of [redacted]. Close valve when ~ 1 mL remains on top to keep column wet. After this step, the columns should not go dry until the completion of loading and rinsing samples.

Appropriately label the SPE cartridges.

Add a poly siphon line to an adapter which has been firmly inserted into the SPE cartridge and place the other end of the line into the corresponding sample container.

Turn on the vacuum and pull the entire sample volume (minimum of 250 mL) through the

cartridge at rate of approximately [redacted].

Stop the sample elution when ~0.1 mL remains. Add ~5 mL of water to the SPE column and restart the elution to complete the loading process. The added water volume ensures there are no small sample droplets remaining that may be clinging to the wall of the SPE cartridge.

After the sample and water rinse has passed through the cartridge, allow the cartridge to completely dry with vacuum (this could take up to 90 minutes). The cartridge should return to a uniform color. NOTE: Remove and replace each cartridge during the drying process to ensure any water droplets that may be in the flow path are eliminated.

10.1.2 SPE Column Wash of Aqueous Samples with Hexane

Add [redacted] to each SPE column and let the column become fully saturated with solvent. Close the stopcock and allow the column to soak for five minutes, then elute to waste.

Load a second [redacted] and elute to waste (without a soaking period).

Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried thoroughly before continuing. The cartridge should return to a uniform color. Wipe any remaining water droplets from the bottom of the stainless steel guide needles using a fresh Kimwipe for each needle prior to proceeding to the next step.

10.1.3 SPE Elution of Aqueous Samples

Note: the use of glass should be avoided where able. However, disposable glass pipettes have a much narrower opening, which is necessary to reduce spillage during the following transfer steps.

Place labeled 15 mL polypropylene test tubes containing [redacted] of Reagent Water as receiving tubes in the SPE manifold.

Rinse the dried sample bottles with [redacted] and transfer to the corresponding SPE cartridge using a disposable glass pipet (NOTE: the sample container has molded ridges in the neck that can trap up to 0.5mL of the solvent rinsate; make sure to tip the container slightly to draw the rinsate out of the ridges). Allow the solution to soak the cartridge for 5 minutes and then elute into the 15 mL collection tube.

Repeat the sample bottle rinse to cartridge elution process with a [redacted] (without the soaking period) The total collection should be approximately 10 mL. Adjust to 10 mL with methanol.

10.1.4 Sample Cleanup with Graphitized Carbon (Optional)

NOTE: If this step is to be performed, do not add the [redacted] to the receiving tubes prior to extract collection. Add [redacted] of graphitized carbon to each sample extract and QC extracts to aid in the removal of organic interferences. Shake vigorously and then let sit for 10 minutes. Centrifuge each sample for 2 minutes at 1000 rpm. Decant the solvent layer into a new 15mL centrifuge tube containing 2 mL of Reagent Water and swirl to mix. Adjust the volume to 10 mL with methanol.

10.1.5 Internal Standard Addition

Add [redacted] internal standard to each extract and vortex to mix well.

Transfer a portion of the extract to a labeled 300µL polypropylene autosampler vial (6 drops or approximately 60µL). Archive the rest of the extract in the event the sample needs re-injection and/or dilution.

Seal the vials with polyethylene screw caps. Note: Teflon lined caps may not be used due to detection of low level concentration of PFAS.

10.2 Soil Sample Preparation

Visually inspect soil samples for homogeneity. Weigh a representative 5 g aliquot of soil, sediment or 1 g of tissue sample into a 50 mL centrifuge tube. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested and enough sample mass is available. Weigh 5 g aliquots of Ottawa sand or 0.1 g of oil for the MB and LCS samples.

Spike the LCS and MS/MSD (if requested) with 25 µL LCS/Matrix Spike Solution. This will result in a sample concentration of 2.0 ng/g (1.0 ng/mL ext).

Add [redacted] of IDA PFC Spiking Solution into each sample and QC sample, for a fixed concentration of 2.5 ng/mL in the final sample vial.

Cap the sample tubes and allow the spikes to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

Add [redacted] to each sample. Cap each sample and shake lightly to confirm container is sealed.

Place all samples in the prep batch into the TCLP tumbler and tumble for 3 hours.

After removing the samples from the tumbler, gently shake each container to confirm the solid material has settled to the bottom of the centrifuge tube, then place in a sonic bath for 12 hours.

Centrifuge each sample at 3500 rpm for 15 minutes.

Transfer the supernate (solvent) to a second, labeled 50 mL centrifuge tube containing 2 mL of Reagent Water.

Slowly add [redacted] to original 50 mL extraction tube. Pour the 2 mL of solvent rinse into the second labeled tube to complete the quantitative transfer.

Place extracts in the ZipVap set to 60 C for ~3 hours with nitrogen flow just strong enough to gently ripple the surface of the extracts. The concentration step is complete when the final volume either gets below 2 mL or maintains at the same level after consecutive checks a 5 minute intervals (this may be due to sample-based moisture contributing to the amount of water in the extract). Remove the sample from the ZipVap when the concentration has completed and allow the extracts to cool.

Adjust the volume of each sample's extract to 15 mL with Reagent Water and add 75 uL of Glacial Acetic Acid to neutralize the solution to pH 6-8. If the extracts contain suspended solids, centrifuge them at 3500 rpm for 15 minutes.

10.2.1 Solid Extract Cleanup by SPE

Condition the SPE cartridges [redacted] by passing the following without drying the column.

Wash with [redacted] with [redacted]. Wash with a second [redacted] followed by a second [redacted]. Close valve when ~ 0.5 mL remains on top to keep column wet. After this step, the columns should not go dry until the completion of loading and rinsing samples.

Appropriately label the SPE cartridges.

Pour each aqueous sample extract into its corresponding SPE cartridge until it is filled. Turn on the vacuum and open the stopcock to load the sample onto the cartridge. Add the remaining extract to the cartridge before it goes dry and stop the flow just before all of the sample has been drawn into the media. [redacted] to the 50 mL centrifuge tube to rinse the tube and complete the quantitative transfer. Pour this rinse into the SPE cartridge and open the stopcock to load the rest of the rinsate onto the cartridge. The added water volume ensures there are no small sample droplets remaining that may be clinging to the wall of the SPE cartridge. Set the centrifuge tubes aside and allow them to completely dry.

After the sample and water rinse has passed through the cartridge, allow the cartridge to completely dry with vacuum (this could take up to 30 minutes). The cartridge should return to a uniform color. NOTE: Remove and replace each cartridge during the drying process to ensure any water droplets that may be in the flow path are eliminated.

10.2.2 SPE Column Wash of Solid Extracts with Hexane

Add [redacted] of hexane to each SPE column and let the column become fully saturated with solvent. Close the stopcock and allow the column to soak for five minutes, then elute to waste.

Load a second [redacted] of hexane and elute to waste (without a soaking period).

Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried thoroughly before continuing. The cartridge should return to a uniform color. Wipe any remaining water droplets from the bottom of the stainless steel guide needles using a fresh Kimwipe for each needle prior to proceeding to the next step.

10.2.3 SPE Elution of Solid Extracts

Place labeled 15 mL polypropylene test tubes containing [redacted] as receiving tubes in the SPE manifold.

Rinse the dried sample tubes with [redacted] and transfer to the corresponding SPE cartridge. Allow the solution to soak the cartridge for 5 minutes and then elute into the 15 mL collection tube.

Repeat sample bottle rinse to cartridge elution process with [redacted] (without the soaking period) The total collection should be approximately 10 mL. Adjust to 10 mL

with methanol.

10.2.4 Sample Cleanup with Graphitized Carbon (Optional)

NOTE: If this step is to be performed, do not add the [redacted] to the receiving tubes prior to extract collection. Add [redacted] of graphitized carbon to each sample extract and QC extracts to aid in the removal of organic interferences. Shake vigorously and then let sit for 10 minutes. Centrifuge each sample for 2 minutes at 1000 rpm. Decant the solvent layer into a new 15mL centrifuge tube containing 2 mL of Reagent Water and swirl to mix. Adjust the volume to 10 mL with methanol.

10.2.5 Internal Standard Addition

Add [redacted] internal standard to each extract and vortex to mix well.

Transfer a portion of the extract to a labeled 300µL polypropylene autosampler vial (6 drops or approximately 60µL). Archive the rest of the extract in the event the sample needs re-injection and/or dilution.

Seal the vials with polyethylene screw caps. Note: Teflon lined caps may not be used due to detection of low level concentration of PFAS.

10.3 Instrument Operating Conditions

Suggested operating conditions are listed below for the [redacted] LCMS system:

Recommended Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 45°C)	Phenomenex Gemini C18 3um, 3.0mm x 100mm				
Mobile Phase Composition	A=20mM Ammonium Acetate (90/10 water/methanol) B=Methanol				
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate mL/min.**
	[redacted]			6	0.60
				6	0.60
				6	0.60
				6	0.60
				6	0.60
				6	0.60
	Maximum pressure limit = 5,000 psi				
Injection Size	[redacted]				
Run Time	[redacted]				
Mass Spectrometer Interface Settings [redacted]					
MS Interface Mode	[redacted]				
Ionspray (volts)	-[redacted]				
Declustering Potential-DP (volts)	[redacted]				
Entrance Potential-EP (volts)	[redacted]				

Source Temp (TEM)	[redacted]
Curtain Gas (CUR)	
Collision Gas (CAD)	
Ion Source Gas 1 (GS1)	
Ion Source Gas 2 (GS2)	
Collision Energy-CE (volts)	
Collision Cell Exit Potential-CXP (volts)	

Recommended Instrument Operating Conditions

Mass Spectrometer Scan Settings [redacted]

Compound	Comments	Reaction (MRM)	Dwell (sec)	DP(v)	EP(v)	CE(v)	CXP(v)
PFBA	Native analyte	212.9 > 169.0	0.011				
13C4 PFBA	IDA	217.0 > 172.0	0.011				
PFPeA	Native analyte	262.9 > 219.0	0.011				
13C5 PFPeA	IDA	267.9 > 223.0	0.011				
PFBS	Native analyte	298.9 > 80.0	0.011				
PFBS_2	Native analyte	298.9 > 99.0	0.011				
13C3 PFBS	IDA	301.9 > 80.0	0.011				
PFHxA	Native analyte	313.0 > 269.0	0.011				
PFHxA_2	Native analyte	313.0 > 119.0	0.011				
13C2 PFHxA	IDA	315.0 > 270.0	0.011				
4:2FTS	Native analyte	327.0 > 307.0	0.011				
M2-4:2FTS	IDA	329.0 > 81.0	0.011				
PFPeS	Native analyte	349.0 > 80.0	0.011				
PFPeS_2	Native analyte	349 > 99.0	0.011				
HFPO-DA	Native analyte	329.1 > 285	0.011				
13C3 HFPO-DA	IDA	332.1 > 287	0.011				
PFHpA	Native analyte	363.0 > 319.0	0.011				
PFHpA_2	Native analyte	363.0 > 169.0	0.011				
13C4 PFHpA	IDA	367.0 > 322.0	0.011				
PFHxS	Native analyte	399.0 > 80.0	0.011				
PFHxS_2	Native analyte	399.0 > 99.0	0.011				
18O2 PFHxS	IDA	403.0 > 84.0	0.011				
DONA	Native analyte	377 > 251	0.011				
DONA_2	Native analyte	377 > 85	0.011				
PFOA	Native analyte	413.0 > 369.0	0.011				
PFOA_2	Native analyte	413.0 > 169.0	0.011				
13C2 PFOA	Internal Std	415.0 > 370.0	0.011				
13C4 PFOA	IDA	417.0 > 372.0	0.011				
6:2FTS	Native analyte	427.0 > 407.0	0.011				
M2-6:2FTS	IDA	429.0 > 81.0	0.011				
PFHpS	Native analyte	449.0 > 80.0	0.011				
PFHpS_2	Native analyte	449.0 > 99.0	0.011				
PFNA	Native analyte	463.0 > 419.0	0.011				
PFNA_2	Native analyte	463.0 > 169.0	0.011				

13C5 PFNA	IDA	468.0 > 423.0	0.011	[redacted]
PFOS	Native analyte	499.0 > 80.0	0.011	
PFOS_2	Native analyte	499.0 > 99.0	0.011	
9Cl-PF3ONS	Native analyte	531 > 351	0.011	
13C4 PFOS	IDA	503.0 > 80.0	0.011	
PFDA	Native analyte	513.0 > 469.0	0.011	
PFDA_2	Native analyte	513.0 > 169.0	0.011	
13C2 PFDA	IDA	515.0 > 470.0	0.011	
8:2FTS	Native analyte	527.0 > 507.0	0.011	
M2-8:2FTS	IDA	529.0 > 81.0	0.011	
PFNS	Native analyte	549.0 > 80.0	0.011	
PFNS_2	Native analyte	549.0 > 99.0	0.011	
MeFOSAA	Native analyte	570 > 419.0	0.011	
d3-MeFOSAA	IDA	573.0 > 419.0	0.011	
11Cl-PF3OUdS	Native analyte	631 > 451	0.011	
FOSA	Native analyte	498.0 > 78.0	0.011	
13C8 FOSA	IDA	506.0 > 78.0	0.011	
PFUdA	Native analyte	563.0 > 519.0	0.011	
PFUdA_2	Native analyte	563.0 > 169.0	0.011	
13C2 PFUdA	IDA	565.0 > 520.0	0.011	
EtFOSAA	Native analyte	584.0 > 419.0	0.011	
d5-EtFOSAA	IDA	589.0 > 419.0	0.011	
PFDS	Native analyte	599.0 > 80.0	0.011	
PFDS_2	Native analyte	599.0 > 99.0	0.011	
PFDoA	Native analyte	613.0 > 569.0	0.011	
PFDoA_2	Native analyte	613.0 > 169.0	0.011	
13C2 PFDoA	IDA	615.0 > 570.0	0.011	
10:2FTS	Native analyte	627 > 607	0.011	
PFDoS	Native analyte	699 > 80	0.011	
PFDoS_2	Native analyte	699 > 99	0.011	
PFTrDA	Native analyte	663.0 > 619.0	0.011	
PFTrDA_2	Native analyte	663.0 > 169.0	0.011	
PFTeDA	Native analyte	713.0 > 669.0	0.011	
PFTeDA_2	Native analyte	713.0 > 169.0	0.011	
13C2 PFTeDA	IDA	715.0 > 670.0	0.011	
PFHxDA	Native analyte	813 > 769	0.011	
PFHxDA_2	Native analyte	813 > 169	0.011	
13C2 PFHxDA	IDA	815 > 770	0.011	
PFODA	Native analyte	913 > 869	0.011	
PFODA_2	Native analyte	913 > 169	0.011	

Recommended Instrument Operating Conditions				
Retention Times & Quantitation ([redacted])				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	[redacted]	13C4 PFBA	[redacted]	Isotope Dilution
PFPeA		13C5 PFPeA		Isotope Dilution
PFBS		13C3 PFBS		Isotope Dilution
4:2FTS		M2-4:2FTS		Internal Standard
PFHxA		13C2 PFHxA		Isotope Dilution
PFPeS		13C3 PFBS		Internal Standard
HFPO-DA		13C3 HFPO-DA		Isotope Dilution
PFHpA		13C4 PFHpA		Isotope Dilution
PFHxS		18O2 PFHxS		Isotope Dilution
DONA		13C4 PFOS		Internal Standard
6:2FTS		M2-6:2FTS		Isotope Dilution
PFOA		13C4 PFOA		Isotope Dilution
PFHpS		13C4 PFOS		Internal Standard
PFNA		13C5 PFNA		Isotope Dilution
PFOS		13C4 PFOS		Isotope Dilution
9Cl-PF3ONS		13C4 PFOS		Internal Standard
8:2FTS		M2-8:2FTS		Isotope Dilution
PFDA		13C2 PFDA		Isotope Dilution
PFNS		13C4 PFOS		Internal Standard
MeFOSAA		d3-MeFOSAA		Isotope Dilution
11Cl-PF3OUdS		13C4 PFOS		Internal Standard
EtFOSAA		d5-EtFOSAA		Isotope Dilution
PFDS		13C4 PFOS		Internal Standard
PFUdA		13C2 PFUdA		Isotope Dilution
FOSA		13C8 FOSA		Isotope Dilution
PFDoA		13C2 PFDoA		Isotope Dilution
10:2FTS		M2-8:2FTS		Internal Standard
PFDoS		13C4 PFOS		Internal Standard
PFTrDA		13C2 PFTeDA		Internal Standard
PFTeDA		13C2 PFTeDA		Isotope Dilution
PFHxDA		13C2 PFHxDA		Isotope Dilution
PFODA		13C2 PFHxDA		Internal Standard

Note: clients must be notified when the quantitation of an analyte is performed using an Internal standard. Changes to these IDA/ISTD associations may be necessary when sources of IDAs are updated: this may include additions as new IDAs become available, or subtractions if IDAs are unavailable.

10.4 Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native and IDA) into the MS/MS electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table above.

10.5 Instrument Calibration

Perform initial calibration with a minimum of five calibration standards before any sample analysis (initial method set-up), whenever a new column is installed, when significant instrument maintenance has been performed, and when the CCV does not meet acceptance criteria. Significant instrument maintenance includes installing a new column, changing the proportioning valve, or changing components of the MS/MS system. A new calibration is not required following minor maintenance.

With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points. The same injection volume must be used for all injections (standards and extracts).

Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by isotope dilution must be < 20% for the curve to be valid.

For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by internal standard (i.e. those compounds that do not have corresponding isotopically labeled analogs) must be < 25% for the curve to be valid.

For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

-Response must increase with increasing concentration.

-The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.

-There should be no carryover at or above 1/2 MRL after a high CAL standard.

-The low cal. point must recover to within 50-150%, and all others must recover to within 70-130%.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.6 Initial Calibration

Prepare the working calibration standards using the recommended formulations given in Appendix B ensuring the lowest calibration standard for each analyte is equal to or below the established RL. Unless otherwise specified on a project basis, use calibration levels 1 to 6 to establish the calibration curve for each analyte.

Prime the instrument by analyzing a minimum of 4 "primer" solutions consisting of 80/20 methanol/water. In general, an HPLC contains components made from PTFE, which enable the pumps to work with many types of organic solvents. Despite efforts to remove as much PTFE as possible, certain components cannot be replaced and contribute PFAS. The longer the system remains idle, the more PFAS that is yielded. Therefore these primers serve to reduce and stabilize the amount of PFAS that are contributed. Immediately following the primers is a Blank, the ICAL sequence (run in ascending order of Level 1 to Level 6), the ICB, the ICV and the first analytical window of extracts (up to 10 field samples). The data is acquired using Sciex's Analyst 1.6.

The Chrom Review data system generates calibration data by generating relative response factors (RRFs) based on the response of the target analyte and its corresponding Isotope Dilution Analyte (or Internal Standard) as well as their injection concentrations to ultimately generate Mean Response Factors. All analytes calibrated using IDA must have RSD values < 20%, all analytes calibrated using ISTD must have RSD values < 25%. The IDA compounds are also calibrated using an external RF model using response and concentration. The IDA RSD must be < 50%. Alternatively, a linear regression curve of concentration vs. peak area for each analyte relative to their corresponding IDA/ISTD and their concentrations calculates the correlation coefficient with 1/concentration weighting. The calibration must have a correlation coefficient (r) $\geq$ 0.995 ($r^2 \geq 0.990$). If criteria are not met, correct the problem and repeat calibration. Further analysis may not proceed without valid calibration.

10.7 Initial Calibration Blank (ICB)

Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of [redacted] fortified with IDA solution at 50 ng/mL

The result for the calibration blank must be less than the reporting limit.

If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.8 Second Source Calibration Verification (ICV)

Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

The recovery for the ICV must meet the appropriate following criteria:

The native analyte must be within or equal to 70-130% for all native analytes quantitated by isotope dilution.

The native analyte must be within or equal to 70-130% for all native analytes quantitated by internal standard (i.e. those compounds that do not have corresponding isotopically labeled analogs).

The IDA recovery must be within or equal to 50-150%.

See Table 3 for corrective actions in the event that the ICV does not meet the criteria above.

10.9 Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid-level range of the curve and should vary throughout the run. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition, the low standard in the curve must be analyzed and must be within ± 50% of the expected value.

The recovery for the CCV standards must be equal to or within 70-130% (50-150% for low level standards) for all natives quantitated by isotope dilution and for all natives quantitated by internal standard. The recovery for the IDA must be within or equal to 70-130% of the true value.

If this is not achieved, the instrument has drifted outside the calibration limits. If the CCV fails again following minor maintenance, the instrument must be recalibrated.

10.10 Isotope Dilution Analytes (IDA)

The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 10.1. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

IDA recoveries are flagged if they are outside of the acceptance limits. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

> Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.
>
> Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.
>
> Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

10.11 Troubleshooting:

Check the following items in case of calibration failures:

Evaluate the failure to determine whether it affects all of the compounds in the ICAL equally. If one ICAL point appears low or high, reprep the curve and rerun, as the error was most likely prep-based. If only a subset of the analytes are affected, check the integration and chromatography to see if there are anomalies; if justifiable, correct the integration so it is consistent with the other ICAL levels.

If there are no peaks for all compounds or no peaks after a specific retention time, ensure that the HPLC pump is pumping properly; it may have shut down due to overpressure or has a leak. If the

pump has shut down, confirm it is primed and replace the in-line filter. If the pressure climbs above expected levels, changing the guard column and even analytical column may be necessary. It's best to chase high pressure sources from the pump forward (ie the post-pump in-line filter, isolator column, post-autosampler in-line filter, guard column, analytical column and MSMS inlet. If the pump is still pumping, check the system pressure. If it is lower than expected, check for leaks. Start with all connections, then move on to pump seals, especially if there are wide variations in pressure when pumping the same solvents at the same flow rates. If the pump is still pumping and the pressure is normal, check to make sure the MSMS is still functioning properly. Most issues with the MSMS system will be noted by the instrument software.

If there are peaks for all analytes, evaluate the peak shapes by comparing them to the ICAL chromatography. If the peaks have changed (shorter and wider), a new guard column may improve peak shape and bring the system back into compliance. If a new column is necessary, a new ICAL will be needed.

Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance
As Needed: Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.
Daily (When in use) Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that multiplier is functioning.

10.12 Sample Analysis

Place the field and QC samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

An example analytical sequence that includes initial calibration (ICAL) is provided below.

Injection Number	Lab Description
1	Primer 1
2	Primer 2
3	Primer 3

Injection Number	Lab Description
4	Primer 4
5	Blank
6	Calibration Level 1
7	Calibration Level 2
8	Calibration Level 3
9	Calibration Level 4 (ICIS)
10	Calibration Level 5
11	Calibration Level 6
12	ICB
13	ICV
14	T-PFOA
15	MB
16	LCS
17-26	(up to) 10 Field samples
27	CCV L4
28-37	(up to) 10 Field samples
38	MS
39	MSD
40	CCV L5
41	MB
42	LCS
43-52	(up to) 10 Field samples
53	CCV L4
54-63	(up to) 10 Field samples
65	MS
66	MSD
67	CCV L5

An example analytical sequence without ICAL:

Injection Number	Lab Description
1	Primer 1
2	Primer 2
3	Primer 3
4	Primer 4
5	CCB
6	CCVL (LOQV)
7	CCVIS (L4)
8	MB
9	LCS
10-19	(up to) 10 Field samples
20	CCV L5
21-30	(up to) 10 Field samples
31	MS
32	MSD
33	CCV L4
34	MB
35	LCS

36-45	(up to) 10 Field samples
46	CCV L5
47-56	(up to) 10 Field samples
57	MS
58	MSD
59	CCV L4

Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and initiate the analytical sequence.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards. The retention times of PFAS with labeled standards must be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the CCVIS standards. *Note: The IS RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.2 Quantitative Identification

The ICAL established in Section 10.10 is used to calculate concentrations for the extracts. The data processing system determines on-column concentration. Final results are calculated by the laboratory's LIMS information system (TALS).

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Check the results of samples analyzed immediately after high concentration samples (those with results above calibration range) for signs of carry-over. Reanalyze all samples suspected of carry -over.

12.3 Calculations

See Appendix C.

12.4 Data Review

Refer to laboratory SOP BR-QA-019 for additional instruction on the requirements for data review. The following sections summarize the general procedure as described in the data review SOP.

12.5 Primary Review

Review the chromatography and quantitation in the data processing system to confirm quantitative and qualitative identification of each target analyte. Perform and document manual integrations only if needed per the instructions in corporate policy CA-Q-S-002, Acceptable Manual Integration Practices.

Upload the data files to TALS and process the batch. Enter job information into the batch editor and add the standards and reagent additions to the worksheet, if necessary. Review the results against acceptance criteria. If acceptance criteria are not met, perform corrective action or make arrangements for corrective action with another analyst.

Set results to primary, secondary, acceptable or rejected. Set results to be reported to a status of primary and secondary. Set results that meet criteria but will not be reported to acceptable. Set results that do not meet criteria to rejected, to prevent inadvertent reporting of data.

Verify that all appropriate QC were performed and acceptable. If insufficient volume is received (MS, MSD, FRB, etc…) document in an NCM. Record all instances where acceptance criteria are not met in a nonconformance memo (NCM).

Verify that all project requirements or program specific requirements were followed. If not, immediately notify the project manager to determine an appropriate course of action. Record decisions made in the data review checklist.

Set the batch to 1st level review. Complete the data review checklist and make arrangements for secondary review by a peer analyst.

12.6 Secondary Data Review (Performed by Peer Analyst)

Record review using the data review checklist.

Verify that all project requirements or program specific requirements were followed. If not, consult with the primary analyst to determine cause. Any decisions made should be recorded on the data review checklist and retained as part of the analytical record.

Review the TALS batch editor to verify ancillary information for the work performed is filled in.

Verify that that the procedures in this SOP were followed. If discrepancy between the SOP and the analytical record is found, consult with the primary analyst to determine the source of the discrepancy. Resolve the discrepancy and verify any modifications to the SOP are properly

documented and were approved by laboratory management. Record all SOP deviations in an NCM.

Spot-check ~15% of samples in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in SOP CA-Q-S-002.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in SOP CA-Q-S-002.

- If an error is suspected, the reviewer must consult with the analyst that performed the integration to determine if a correction is necessary. Input from the Technical Manager (TM), Department Manager (DM), or QA Manager (QAM) may be sought as necessary. **The reviewer may not reintegrate except in those circumstances approved by laboratory management**, such as when the analyst that performed the integration is on vacation. If re-integration is performed by the reviewer, the reviewer is now considered the "primary analyst" and the re-integration is subject to the same review and documentation requirements as the original integration.

Verify acceptance criteria were met. If not, verify that corrective actions were performed and the nonconformance was documented with an NCM. Review the NCM to verify the form is filled out and the requisite information has been included in the internal comments tab. If corrective action was not performed and the failure not documented, consult with the primary analyst to determine cause. Consult with the primary analyst and department management to determine what actions should be taken, then follow-through with the decision made.

Run the QC checker and fix any problems found. Run and review the deliverable for gross error such as missing data. Fix any problems found.

When review is complete set the method chain to lab complete. Complete the data review checklist and forward associated paperwork to report/project management.

12.7 Data Reporting & Record Retention

The specifications for data reporting are set by the project manager and are performed by TALS using the formatter selected by the PM. The type of deliverable is also set by the PM based on various deliverable options in the TALS system. The formatters and deliverables are programmed into TALS by corporate IT staff and cannot be modified locally.

The following sections describe the default reporting scheme set for this method in TALS:

Data is retained, managed and archived as specified in laboratory SOP BR-QA-014 Laboratory Records.

13.0 Method Performance

13.1 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. An initial method detection limit study is performed in accordance with SOP BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample can be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP BR-QA-011.

14.0 Pollution Control

It is Test America's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.0 Waste Management

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

- Vials containing sample extracts: Satellite Container: 30 gallon poly barrel located under GC-Semi prep hood.
- Solvent Waste: Satellite Container: 5 gallon poly carboy located under LCMSMS.

16.0 References / Cross References

- Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, "Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and 19FNMR," Analytical Chemistry 2001, 73, 2200-2206.
- John Giesy et al., "Accumulation of Perfluorooctane Sulfonate in Marine Mammals", Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.
- U.S. EPA, "Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method", EPA 712-C-95-174, August 1995.
- STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanoate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.
- STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanoate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.
- STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.
- Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.
- Method ISO 25101, "Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry", First Edition, 2009-03-01, International Organization for Standardization, Technical Committee ISO/TC 147, Water Quality, Subcommittee SC 2, Physical, chemical and biochemical methods.
- US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092.
- Laboratory SOP BR-QA-005 *Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).*
- Laboratory SOP BR-QA-011 *Employee Training*
- Laboratory SOP BR-EH-001 *Hazardous Waste*
- Laboratory SOP BR-QA-014 *Laboratory Records*
- Laboratory SOP BR-QA-006 *Procedures & Documentation Requirements for Manual Integration*
- Laboratory Quality Assurance Manual (QAM)
- Corporate TestAmerica SOP CA-Q-S-002 *Manual Integrations*.

17.0 Method Modifications

Modification Number	Method Reference	Modification & Technical Justification
1	Section 7.2	Method 25101 specifies that the values reported for PFOA and PFOS shall be the linear isomer only. In keeping with the dictates of USEPA 537 and other US conventions, the laboratory reports both the branched (when present) and linear isomers as a single value for these compounds.
2	Section 10.1	A different SPE cartridge, Waters OASIS WAX, is used for the extraction process. As a result, solvents and elution procedures are different.
3	Section 10.1	The samples are fortified with a greater number of labeled analytes (most analytes have labeled versions) prior to extraction.
4	Section 10.5	The HPLC Column, Eluents and gradient conditions have changed.
5	Section 10.5	For non-drinking water matrices, the analyte list has expanded. The number of labeled analytes has also expanded to improve quantitation.
6	Table 1	The reporting limits have changed to a consistent value.
7	Appendix B	Calibration levels have been changed so all levels have the same analyte concentration.

18.0 Attachments

- Table 1: Routine Compound List and LOQ
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Table 4: Control Limits
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

19.0 Revision History (all revision history must be retained in this SOP)

Revision 6.0

- Updated cover page dates, copyright information, and signatories
- Throughout: Added support for soil, sediment and tissue matrices.
- Throughout: Removed reference to analysis using Waters instrumentation.
- Throughout: Removed reference to final extract concentration for aqueous samples.
- Section 1.1: Update Fluorotelomer sulfonates (FTS) to report acid forms
- Section 11.0: Added corrective action requirement as it is a corporate requirement to include.

Revision 5.0

- Updated cover page dates, copyright information, and signatories
- Throughout: removed references to drinking water. Will add back if adopted.
- Throughout: removed solid extraction/analysis verbiage missed in previous revision.
- Throughout: revised formatting to be consistent
- Throughout: added PFHxDA, PFODA, PFDoS, 10:2FTS, HFPO_DA, DONA, F53BMajor, and F53B Minor as additional analytes and IDAs
- Section 4.0: added interference information about $^{13}C_2$-PFHxDA
- Section 6.1: updated to include additional laboratory apparatus information
- Section 6.2: updated to include additional instrument and more detail for existing instrument
- Section 7.1: added more detail to reagent information and the addition of Ammonium acetate and Ammonium hydroxide
- Section 7.2: added PFHpS and PFDS as other PFAS not available in the acid form. Added the IDA and ISTD are added at a fixed concentration and removed the low level reference
- Section 9.1 added a NCM must be added for MS/MSD
- Section 10.1: removed the low level spike reference and added the PFAS-IDA solution is added to each sample and QC sample in concentrated extract and non-concentrated extracts
- Section 10.2: In the previous version of this SOP, the “Note” was removed and replaced with “Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect All glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.”
- Section 10.2: changed wording to clarify addition of poly siphon line into the SPE cartridge
- Section 10.3: removed to keep test tube as keep and added “Note: If the extracts will note be concentrated, use [redacted] for the second bottle rinse so the final volume is approximately 8mL.”
- Section 10.5: added sample cleanup with graphitized carbon section
- Section 10.6: added wording to have [redacted] of reagent water to the 10mL extract at this time
- Section 10.7: updated wording
- Section 10.8: added operating system for new instrument and added more detail for existing instrument
- Section 10.17: updated sample analysis to include calibration currently in use
- Table 1 and Table 4: updated to include additional analytes and IDAs
- Appendix A: updated terms and definitions from body of SOP
- Appendix B: updated to include additional analytes and IDAs

Revision 4.0

- Updated cover page dates, copyright information, and signatories
- Headers: removed TestAmerica logo and added Eurofins logo
- Throughout: removed references to drinking water. Will add back if adopted.
- Throughout: revised formatting to be consistent
- Section 1.1: added note about addition of Appendix D, removed NJDEP as PAB
- Section 10.1.3: added note about the use of glass pipettes

- Section 10.3: In a previous version of this SOP, Table “Recommended Instrument Operating Conditions” incorrectly referenced PFTrDA as Isotope Dilution, so this was corrected to Internal Standard and added note to contact clients for ISTD quantitation.
- Removed verbiage regarding soil LOQ from Note on Table 1.
- Added Appendix D: NJDEP secondary certified analytes list

Revision 3.0

- Updated cover page dates and signatories
- Section 10.1: added note for handling incomplete volume extraction process
- Section 18: added previous revision history back into SOP
- Throughout: updated QC criteria from EPA 537 r1.1 that was missed in previous revision
- Throughout: removed solid extraction/analysis verbiage missed in previous revision.
- Throughout: updated calibration to include criteria from EPA 537 r1.1 and to include the 9 calibration points currently in use.
- Throughout: minor formatting updates

Rev 2.1:

- Updated cover page dates and signatories
- Section 8: added preservation requirements for DW samples.
- Throughout: updated QC criteria to match EPA537 rev1.1
- Throughout: removed references to solid and tissue extraction/analysis.

Rev 2.0

- Updated cover page and signatories
- Section 8: added preservation requirements for DW samples.
- Throughout: included verbiage that Non-drinking water matrices are not certified under PAB.
- Throughout: separated DW and non-DW limits and QC requirements.
- Throughout: minor formatting and typographical corrections.
- Tables 3 & 4: updated limit to meet EPA 537 criteria.
- Appendix A: updated terms and definitions from body of SOP

Rev 1.0

- Extended analyte list to 21 native compounds and 18 IDAs.
- Altered concentration step in extract preparation by employing a reagent water keeper instead of concentrating to dryness.
- Incorporated use of internal standard for IDA recovery calculation.

Revision 0.0: 05/19/2017

- New SOP based on USEPA method 537

Previous revisions are retained by the QA department.

Table 1: Routine Compound List & Limit of Quantitation (LOQ)

Compound Name	Abbreviation	CAS #	Water (ng/L)	Soil/ Sediment (ug/Kg)	Tissue (ug/Kg)
Perfluoroalkylcarboxylic acids (PFCAs)					
Perfluoro-n-butanoic acid	PFBA	375-22-4	2.0	0.20	1.0
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3	2.0	0.20	1.0
Perfluoro-n-hexanoic acid	PFHxA	307-24-4	2.0	0.20	1.0
Perfluoro-n-heptanoic acid	PFHpA	375-85-9	2.0	0.20	1.0
Perfluoro-n-octanoic acid	PFOA	335-67-1	2.0	0.20	1.0
Perfluoro-n-nonanoic acid	PFNA	375-95-1	2.0	0.20	1.0
Perfluoro-n-decanoic acid	PFDA	335-76-2	2.0	0.20	1.0
Perfluoro-n-undecanoic acid	PFUdA	2058-94-8	2.0	0.20	1.0
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1	2.0	0.20	1.0
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8	2.0	0.20	1.0
Perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7	2.0	0.20	1.0
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5	2.0	0.20	1.0
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6	2.0	0.20	1.0
Perfluorinated sulfonic acids (PFSAs)					
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5	2.0	0.20	1.0
Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-4	2.0	0.20	1.0
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4	2.0	0.20	1.0
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8	2.0	0.20	1.0
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1	2.0	0.20	1.0
Perfluoro-1-nonanesulfonic acid	PFNS	68259-12-1	2.0	0.20	1.0
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3	2.0	0.20	1.0
Perfluoro-1-dodecanesulfonic acid	PFDoS	79780-39-5	2.0	0.20	1.0
Perfluorinated sulfonamides (FOSA)					
Perfluoro-1-octanesulfonamide	FOSA	754-91-6	2.0	0.20	1.0
Perfluorinated sulfonamidoacetic acids (FOSAA)					
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6	20.0	2.0	10.0
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9	20.0	2.0	10.0
Fluorotelomer sulfonates (FTS)					
1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*	4:2 FTS	757124-72-4	20.0	2.0	10.0
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2	20.0	2.0	10.0
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4	20.0	2.0	10.0
1H,1H,2H,2H-perfluorododecane sulfonate*(10:2)*	10:2 FTS	120226-60-0	20.0	2.0	10.0
Fluorinated Replacement Chemicals					
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	4.0	0.40	2.0
4,8-dioxa-3H-perfluorononanoic acid	DONA	919005-14-4	2.0	0.20	1.0
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	F53B Major (9Cl-PF3ONS)	756426-58-1	2.0	0.20	1.0
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	F53B Minor (11Cl-PF3OUdS)	763051-58-1	2.0	0.20	1.0

NOTE: The LOQ values may vary. The Water LOQ is based on a 250mL nominal sample volume.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide (3-0-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. 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 damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	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 (3-0-1)	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol (2-3-0)	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.
Potassium Hydroxide (3-0-1)	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Potassium Persulfate (2-0-1-OX)	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.

Table 3: QC Summary, Acceptance Criteria and Recommended Corrective Action (EPA537)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6-Point Calibration (5 point minimum for CF and Linear Regression) (ICAL)	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF = RSD $\leq$ 20% (compounds calibrated via IDA) CF = RSD $\leq$ 25% (compounds calibrated using "near-IDA" compounds) CF = RSD $\leq$ 50% (IDA standards using ISTD) Each cal pt. = +/-30%Rec. (+/-50%Rec for cal low pt.) Linear Regression: $r^2 \geq 0.990$	Correct problem and repeat initial calibration.
IDA Response	Every injection contains the IDA analytes	Non-DW matrices: Standards: 50-150% recovery Field samples: 50-150% recovery (poor responding IDAs: 25-150%) (reportable if >10x S/N ratio and >10% ICAL RF)	Standard failures must be investigated to determine the cause of the failure. Recalibration may be required. Samples with recoveries outside acceptance limits must be evaluated for data usability. Re-extraction may be necessary if data quality has been adversely affected.
IS Response	Every injection contains the IS analyte	ICAL Standards: Area of individual points must not deviate by more than 50% of ICAL mean area response Samples following ICAL: 50-150% of ICAL mean response Ongoing CCV: 50-150% of ICAL mean response Post-CCV Samples: Area must be within 50-150% of most recent CCVIS (daily opening CCV)	Standard failures must be investigated to determine the cause of the failure. Recalibration may be required. Sample failures may be matrix related and should be evaluated to determine if the data quality has been adversely affected.
Initial Calibration Blank (ICB)	Immediately following the ICAL	Non-DW: < RL for all target analytes	Determine source of interference/contamination, eliminate it and recalibrate.
Second Source Standard Verification (ICV)	Prior to the analysis of samples. Generally immediately after the ICB.	+/-30 for analytes, IS, and SUR.	Correct problem and verify second source standard. If that fails, repeat calibration.
Continuing Calibration Verification (CCV)	Beginning of each analytical sequence, every ten field samples and at the end of each analytical sequence. Alternate between levels 3, 4 and 5.	+/-30%	Rerun any samples analyzed before and after the failing CCV. Take corrective action; if subsequent CCV analyses fail, recalibrate instrument.
Continuing Calibration Verification-Low (CCVL)	Beginning of each analytical sequence that is not preceded by an ICAL to show LOQ is still valid.	CF = 50-150% (ISTD targets) IDA 50-150%	Stop sample acquisition. Take corrective action; if subsequent CCV analyses fail, recalibrate instrument.
Method Blank	One per extraction batch of 20 or fewer samples	Non-DW: < RL for all target analytes	Reprocess MB and associated samples if any target analyte in the MB is at or above the RL, greater than 1/10 the amount detected in any sample or 1/10 the regulatory limit, whichever is greater. If the target is not greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with appropriate qualifiers. If insufficient sample is available to reprocess, report data with appropriate qualifiers.
Laboratory Control Sample	One per extraction batch of 20 or fewer samples (rotate between Low, Med, High)	%R within control limits. See Table 4	Reprep and reanalyze samples for failed analytes. If reanalysis is not possible due to insufficient sample volume, report data with appropriate data qualifiers.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike / Matrix Spike Duplicate	One set per extraction batch when sufficient sample volume is provided	%R within control limits. See Table 4	Evaluate to determine if there is a matrix effect or analytical error. If analytical error, reanalyze or reprocess as appropriate.
Sample Duplicate	One per extraction batch of 20 or fewer samples	RPD within control limits. See Table 4	Evaluate data to determine source for error. If analytical error is suspected, reanalyze or reprocess as appropriate.
Field Reagent Blank	Per client sample set	Non-DW: < RL for all target analytes	Analysis only required if samples contain target analytes at or above the RL. If analytes are present in the FRB at >1/3 RL, all samples must be recollected and re-analyzed.

Table 4: LCS and MS/MSD Control Limits*

Analyte	Water (Low Level) %R	Water (Med-High Level) %R	RPD
Perfluorobutanoic acid (PFBA)	50-150	70-130	20
Perfluoropentanoic acid (PFPeA)	50-150	70-130	20
Perfluorobutanesulfonic acid (PFBS)	50-150	70-130	20
Perfluorohexanoic acid (PFHxA)	50-150	70-130	20
Perfluoropentanesulfonic acid (PFPeS)	50-150	70-130	20
Perfluoroheptanoic acid (PFHpA)	50-150	70-130	20
Perfluorohexanesulfonic acid (PFHxS)	50-150	70-130	20
Perfluorooctanoic acid (PFOA)	50-150	70-130	20
Perfluoroheptanesulfonic acid (PFHpS)	50-150	70-130	20
Perfluorononanoic acid (PFNA)	50-150	70-130	20
Perfluorooctanesulfonic acid (PFOS)	50-150	70-130	20
Perfluorodecanoic acid (PFDA)	50-150	70-130	20
Perfluorononanesulfonic acid (PFNS)	50-150	70-130	20
Perfluoroundecanoic acid (PFUdA)	50-150	70-130	20
Perfluorodecanesulfonic acid (PFDS)	50-150	70-130	20
Perfluorooctanesulfonamide (FOSA)	50-150	70-130	20
Perfluorododecanoic acid (PFDoA)	50-150	70-130	20
Perfluorododecanesulfonic acid (PFDoS)	50-150	70-130	20
Perfluorotridecanoic acid (PFTrDA)	50-150	70-130	20
Perfluorotetradecanoic acid (PFTeDA)	50-150	70-130	20
Perfluorohexadecanoic acid (PFHxDA)	50-150	70-130	20
Perfluorooctadecanoic acid (PFODA)	50-150	70-130	20
1H,1H,2H,2H Perfluorohexanesulfonate (4:2FTS)	50-150	70-130	20
1H,1H,2H,2H Perfluorooctanesulfonate (6:2FTS)	50-150	70-130	20
1H,1H,2H,2H Perfluorodecanesulfonate (8:2FTS)	50-150	70-130	20
1H,1H,2H,2H Perfluorododecanesulfonate (10:2FTS)	50-150	70-130	20
N-Methyl Perfluorooctane sulfonamidoacetic acid (N-MeFOSAA)	50-150	70-130	20
N-Ethyl Perfluorooctane sulfonamidoacetic acid (N-EtFOSAA)	50-150	70-130	20
Hexafluoropropylene oxide dimer acid	50-150	70-130	20
4,8-dioxa-3H-perfluorononanoic acid	50-150	70-130	20
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	50-150	70-130	20
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	50-150	70-130	20

**The limits in this table are those in effect as of the published date of this SOP. The %R limits are specified by EPA 537r1.1 in sections 9.33, 9.36, and 9.37. The RPD the lab uses is more strict than those referenced in EPA 537 r1.1. If the lab makes changes to any of these limits, the updated limits will be no less strict than those specified in EPA537.*

Appendix A: Terms and Definitions

PFCAs: Perfluorocarboxylic acids
PFSAs: Perfluorinated sulfonic acids
FOSA: Perfluorinated sulfonamide
PFOA: Perfluorooctanoic acid
PFOS: Perfluorooctane sulfonate
PTFE: Polytetrafluoroethylene (e.g., Teflon®)
SPE: Solid phase extraction**.**
PP: Polypropylene
PE: Polyethylene
HDPE: High density polyethylene
AFFF: Aqueous Film Forming Foam
IDA: Isotope dilution analytes
Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).
Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.
Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.
Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.
Calibration Standard: A substance or reference used to calibrate an instrument.
Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.
Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.
Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)
Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.
Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.
Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.
Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.
Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.
Matrix Spike Duplicate (MSD): a second replicate matrix spike
Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).
Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.
Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.
Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.
Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.
Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.
Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.
Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.
Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in methanol using Class A volumetric glassware and Hamilton syringes and assign an expiration date of 1 year from date of preparation unless the parent standard expires sooner; then use the earlier date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance. For stock standards solutions made from neat material, assign an expiration date of 2 years from the date of formulation.

Stock Standard Solutions

PFAS LCS/Matrix Spike Solution 1000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
PFBA	Wellington Laboratories Code: PFBA	Perfluorobutanoic acid	50	200	10	1000
PFPeA	Wellington Laboratories Code: PFPeA	Perfluoropentanoic acid	50	200		1000
PFBS	Wellington Laboratories Code: L-PFBS	Perfluorobutanesulfonic acid	44.2	200		884
PFHxA	Wellington Laboratories Code: PFHxA	Perfluorohexanoic acid	50	200		1000
PFPeS	Wellington Laboratories Code: L-PFPeS	Perfluoropentanesulfonic acid	46.9	200		938
PFHpA	Wellington Laboratories Code: PFHpA	Perfluoroheptanoic acid	50	200		1000
PFHxSK	Wellington Laboratories Code: br-PFHxSK	Perfluorohexanesulfonic acid	45.5	200		910
PFOA	Wellington Laboratories Code: PFOA	Perfluorooctanoic acid	50	200		1000
PFHpS	Wellington Laboratories Code: L-PFHpS	Perfluoroheptanesulfonic acid	47.6	200		952
PFNA	Wellington Laboratories Code: PFNA	Perfluorononanoic acid	50	200		1000
PFOS	Wellington Laboratories Code: br-PFOSK	Perfluorooctanesulfonic acid	46.4	200		928
PFDA	Wellington Laboratories Code: PFDA	Perfluorodecanoic acid	50	200		1000
PFNS	Wellington Laboratories Code: L-PFNS	Perfluorononanesulfonic acid	48.0	200		960
PFUdA	Wellington Laboratories Code: PFUdA	Perfluoroundecanoic acid	50	200		1000
PFDS	Wellington Laboratories Code: L-PFDS	Perfluorodecanesulfonic acid	48.2	200		964
FOSA	Wellington Laboratories Code: FOSA-I	Perfluorooctane sulfonamide	50	200		1000
PFDoA	Wellington Laboratories Code: PFDoA	Perfluorododecanoic acid	50	200		1000
PFDoS	Wellington Laboratories Code: L-PFDoS	Perfluorododecanesulfonic acid	48.4	200		968
PFTrDA	Wellington Laboratories Code: PFTrDA	Perfluorotridecanoic acid	50	200		1000
PFTeDA	Wellington Laboratories Code: PFTeDA	Perfluorotetradecanoic acid	50	200		1000
PFHxDA	Wellington Laboratories Code: PFHxDA	Perfluorohexadecanoic acid	50	200		1000
PFODA	Wellington Laboratories Code: PFODA	Perfluorooctadecanoic acid	50	200		1000
4:2FTS	Wellington Laboratories Code: 4:2FTS	1H,1H,2H,2H-perfluorohexane sulfonate (4:2)	46.7	200		934
6:2FTS	Wellington Laboratories Code: 6:2FTS	1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	47.4	200		948

8:2FTS	Wellington Laboratories Code: 8:2FTS	1H,1H,2H,2H-perfluorodecane sulfonate (8:2)	47.9	200		958
10:2FTS	Wellington Laboratories Code: 10:2FTS	1H,1H,2H,2H-perfluorododecane sulfonate (10:2)	48.2	200		964
NMeFOSAA	Wellington Laboratories Code: br-NMeFOSAA	N-methyl Perfluorooctane sulfonamidoacetic acid	50	200		1000
NEtFOSAA	Wellington Laboratories Code: br-NEtFOSAA	N-ethyl Perfluorooctane sulfonamidoacetic acid	50	200		1000
HFPO-DA	Wellington Laboratories Code: HFPO-DA	Hexafluoropropylene oxide dimer acid	50	200		1000
DONA	Wellington Laboratories Code: NaDONA	4,8-dioxa-3H-perfluorononanoic acid	47.1	200		942
9Cl-PF3ONS	Wellington Laboratories Code: 9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanone-1-sulfonate	46.6	200		932
11Cl-PF3OUdS	Wellington Laboratories Code: 11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonate	47.1	200		942

Solvent: Methanol

PFAS-IDA Solution (Surrogate) 1000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C4 PFBA	Wellington Laboratories Code: MPFBA	$^{13}C_4$-Perfluorobutanoic acid	50	200	10	1000
13C5-PFPeA	Wellington Laboratories Code: MPFPeA	$^{13}C_5$-Perfluoropentanoic acid	50	200		1000
13C3-PFBS	Wellington Laboratories Code: M3PFBS	$^{13}C_3$-Perfluorobutanesulfonic acid	46.5	200		930
13C2 PFHxA	Wellington Laboratories Code: MPFHxA	$^{13}C_2$-Perfluorohexanoic acid	50	200		1000
13C4 PFHpA	Wellington Laboratories Code: M4PFHpA	$^{13}C_4$-Perfluoroheptanoic acid	50	200		1000
18O2 PFHxS	Wellington Laboratories Code: MPFHxS	$^{18}O_2$-Perfluorohexanesulfonic acid	47.3	200		946
13C4 PFOA	Wellington Laboratories Code: MPFOA	$^{13}C_4$-Perfluorooctanoic acid	50.0	200		1000
13C5 PFNA	Wellington Laboratories Code: MPFNA	$^{13}C_5$-Perfluorononanoic acid	50.0	200		1000
13C4 PFOS	Wellington Laboratories Code: MPFOS	$^{13}C_4$-Perfluorooctanesulfonic acid	47.8	200		956
13C2 PFDA	Wellington Laboratories Code: MPFDA	$^{13}C_2$-Perfluorodecanoic acid	50.0	200		1000
13C8 FOSA	Wellington Laboratories Code: M8FOSA-I	$^{13}C_8$-Perfluorooctane sulfonamide	50.0	200		1000
13C2 PFUdA	Wellington Laboratories Code: MPFUdA	$^{13}C_2$-Perfluoroundecanoic acid	50.0	200		1000
13C2 PFDoA	Wellington Laboratories Code: MPFDoA	$^{13}C_2$-Perfluorododecanoic acid	50.0	200		1000
13C2 PFTeDA	Wellington Laboratories Code: MPFTeDA	$^{13}C_2$-Perfluorotetradecanoic acid	50.0	200		1000
13C2 PFHxDA	Wellington Laboratories Code: MPFHxDA	$^{13}C_2$-Perfluorohexadecanoic acid	50.0	200		1000
M2-4:2FTS	Wellington Laboratories Code: M2-4:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-hexane sulfonate (4:2)	46.7	200		934
M2-6:2FTS	Wellington Laboratories Code: M2-6:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-octane sulfonate (6:2)	47.5	200		950
M2-8:2FTS	Wellington Laboratories Code: M2-8:2FTS	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-decane sulfonate (8:2)	47.9	200		958
d3-NMeFOSAA	Wellington Laboratories Code: d3-M-MeFOSAA	N-methyl-d_3-perfluoro-1-octane sulfonamidoacetic acid	50.0	200		1000
d5-NEtFOSAA	Wellington Laboratories Code: d5-M-EtFOSAA	N-ethyl-d_5-perfluoro-1-octane sulfonamidoacetic acid	50.0	200		1000

M3HFPO-DA	Wellington Laboratories Code: M3HFPO-DA	$^{13}C_3$-Hexafluoropropylene oxide dimer acid	50.0	200		1000

Solvent: Methanol

PFAS Internal Standard Stock Solution 5000 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C2 PFOA	Wellington Laboratories Code: M2PFOA	$^{13}C_2$-Perfluorooctanoic acid	50.0	400	4	5000

Solvent: Methanol

PFAS Internal Standard Spiking Solution 2500 ng/mL

Parent Standard	Vendor	Component	Stock Standard Conc (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc (ng/mL)
13C2 PFOA	Wellington Laboratories Code: M2PFOA	$^{13}C_2$-Perfluorooctanoic acid	50.0	200	4	2500

Solvent: Methanol

PFAS-IDA-IS Calibration Standards Level 1-Level 6

ICAL Level	Vol of PFAS LCS/Matrix Spike (µL)	Nominal Conc of PFAS (ng/mL)	Vol of PFAS-IDA Solution (µL)	Conc of IDA (ng/mL)	Vol of 5ppm PFAS-IS Stock Solution (µL)	Conc of IS (ng/mL)	Vol of Water (µL)	Vol of 80/20 MeOH/H2O (µL)	Final Vol (mL)
1	4	1.0	200	50	40	50	51	3745	4.0
2	2	2.0	50	50	10	50	13	935	1.0
3	16	5.0	160	50	32	50	44	2980	3.2
4	72	20.0	180	50	36	50	63	3285	3.6
5	160	50.0	160	50	32	50	80	2800	3.2
6	240	200	60	50	12	50	75	825	1.2

The solvent is 80/20 Methanol/Water.

Appendix C: Equations

Initial Calibration Curve Evaluation:

The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
b = slope
c = intercept

The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

The external standard method uses the following equation:

Equation 3

$$ResponseFactor = \frac{Peak\ Area}{Concentration\ of\ Solution (ng/mL)}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 5

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

Water Sample Result Calculation:

Equation 6

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

IDA Recovery Calculation:

Equation 8

$$\%\,\mathrm{Recovery} = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X 100$$

Where ng/g = µg/kg and:

RF_{IDA} = Response Factor for IDA compound
A_t = Area response for IDA compound
A_{IS} = Area Response for IS compound
Q_{IS} = Amount of IS added
Q_t = Amount of IDA added

Calibration Factor (CF_x) = Peak area or height $_{(x)}$ / Standard concentration $_{(\mu g/L)}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = Calculated Concentration – Theoretical Concentration / Theoretical Concentration x 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$C_{extract}$ (µg/L) = $\frac{\text{Peak Area (or Height)}}{\overline{CF}}$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Appendix D: Analytes applied for Secondary Certification with NJDEP

Compound Name	Abbreviation	CAS #
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUdA (PFUnA)	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA (PFTA)	376-06-7
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorooctanesulfonic acid	PFOS	1763-23-1

NEW YORK STATE OF OPPORTUNITY. Department of Health

ANDREW M. CUOMO
Governor

HOWARD A. ZUCKER, M.D., J.D.
Commissioner

SALLY DRESLIN, M.S., R.N.
Executive Deputy Commissioner

LAB ID: 10391

April 01, 2020

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE
SUITE #11
SOUTH BURLINGTON, VT 05403

Certificate Expiration Date:
April 01, 2021

Dear Ms. Dusablon,

Enclosed are certificate(s) of approval issued to your environmental laboratory for the current permit year. The certificate(s) supersede(s) any previously issued one(s) and is(are) in effect through the expiration date listed. Please carefully examine the certificate(s) to insure that the categories, subcategories, analytes, and methods for which your laboratory is approved are correct. In addition, verify that your laboratory's name, address, lead technical director, and identification number are accurate.

Pursuant to NYCRR Subpart 55-2.2, original certificates must be posted conspicuously in the laboratory and copies shall be made available to any client of the laboratory upon request.

Pursuant to NYCRR Subpart 55-2.6, any misrepresentation of the fields of accreditation (category - method - analyte) for which your laboratory is approved may result in denial, suspension, or revocation of your certification. Any use of the Environmental Laboratory Approval Program (ELAP) or National Environmental Laboratory Accreditation Program (NELAP) name, reference to the laboratory's approval status, and/or using the NELAP logo in any catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports, or other materials must include the laboratory's ELAP identification number and distinguish between testing for which the laboratory is approved and testing for which the laboratory is not approved.

If you have any questions, please contact us at the Environmental Laboratory Approval Program, Wadsworth Center, New York State Department of Health, Empire State Plaza, Albany NY, 12237; by phone at (518) 485-5570; by facsimile at (518) 485-5568; and by email at elap@health.ny.gov.

Sincerely,

Victoria Pretti

Victoria Pretti
Director and QA Officer
Environmental Laboratory Approval Program

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

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:

Miscellaneous

Perchlorate	EPA 331.0

Serial No.: 61075

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES POTABLE WATER
All approved subcategories and/or analytes are listed below:

Perfluorinated Alkyl Acids

Perfluorooctanesulfonic acid (PFOS)	ISO 25101
Perfluorooctanoic acid (PFOA)	ISO 25101

Serial No.: 61076

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

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:

Dissolved Gases

Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175

Metals I

Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D
	EPA 6020B
Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D

Metals I

Potassium, Total	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D

Metals II

Aluminum, Total	EPA 6010D
	EPA 6020B
Arsenic, Total	EPA 6010D
	EPA 6020B
Beryllium, Total	EPA 6010D
	EPA 6020B
Mercury, Total	EPA 7470A
Selenium, Total	EPA 6010D
	EPA 6020B
Vanadium, Total	EPA 6010D
	EPA 6020B
Zinc, Total	EPA 6010D
	EPA 6020B

Metals III

Cobalt, Total	EPA 6010D
	EPA 6020B
Molybdenum, Total	EPA 6010D
	EPA 6020B
Thallium, Total	EPA 6010D
	EPA 6020B

Serial No.: 61077

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

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:

Metals III

Tin, Total	EPA 6010D
Titanium, Total	EPA 6010D

Miscellaneous

Perchlorate	EPA 6850

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8330B
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 8330B
2,6-Dinitrotoluene	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A

Polychlorinated Biphenyls

Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A

Sample Preparation Methods

EPA 3010A
EPA 3510C
EPA 3520C

Serial No.: 61077

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NELAP RECOGNIZED TNI ACCREDITATION BODY

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Characteristic Testing	
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311
Metals I	
Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D
	EPA 6020B
Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D
	EPA 6020B
Metals I	
Silver, Total	EPA 6010D
	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D
Metals II	
Aluminum, Total	EPA 6010D
	EPA 6020B
Antimony, Total	EPA 6010D
	EPA 6020B
Arsenic, Total	EPA 6010D
	EPA 6020B
Beryllium, Total	EPA 6010D
	EPA 6020B
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010D
	EPA 6020B
Vanadium, Total	EPA 6010D
	EPA 6020B
Zinc, Total	EPA 6010D
	EPA 6020B
Metals III	
Cobalt, Total	EPA 6010D
	EPA 6020B
Molybdenum, Total	EPA 6010D

Serial No.: 61078

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Metals III	
Molybdenum, Total	EPA 6020B
Thallium, Total	EPA 6010D
	EPA 6020B
Tin, Total	EPA 6010D
Titanium, Total	EPA 6010D
Miscellaneous	
Organic Carbon, Total	USER DEFINED Lloyd Kahn mod
Perchlorate	EPA 6850
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8330B
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 8330B
2,6-Dinitrotoluene	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B

Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Sample Preparation Methods	
	EPA 3050B
	EPA 3550C
	EPA 3540C
	EPA 3541

Serial No.: 61078

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:

Acrylates	
Acetonitrile	EPA TO-15
Methyl methacrylate	EPA TO-15
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA TO-15
Hexachlorobutadiene	EPA TO-15
Polychlorinated Biphenyls	
PCBs and Aroclors	EPA TO-10A
	EPA TO-4A
Polynuclear Aromatics	
Acenaphthene	EPA TO-13A
Acenaphthylene	EPA TO-13A
Anthracene	EPA TO-13A
Benzo(a)anthracene	EPA TO-13A
Benzo(a)pyrene	EPA TO-13A
Benzo(b)fluoranthene	EPA TO-13A
Benzo(g,h,i)perylene	EPA TO-13A
Benzo(k)fluoranthene	EPA TO-13A
Chrysene	EPA TO-13A
Dibenzo(a,h)anthracene	EPA TO-13A
Fluoranthene	EPA TO-13A
Fluorene	EPA TO-13A
Indeno(1,2,3-cd)pyrene	EPA TO-13A
Naphthalene	EPA TO-13A
	EPA TO-15

Polynuclear Aromatics	
Phenanthrene	EPA TO-13A
Pyrene	EPA TO-13A
Purgeable Aromatics	
1,2,4-Trimethylbenzene	EPA TO-15
1,2-Dichlorobenzene	EPA TO-15
1,3,5-Trimethylbenzene	EPA TO-15
1,3-Dichlorobenzene	EPA TO-15
1,4-Dichlorobenzene	EPA TO-15
2-Chlorotoluene	EPA TO-15
Benzene	EPA TO-15
Chlorobenzene	EPA TO-15
Ethyl benzene	EPA TO-15
m/p-Xylenes	EPA TO-15
o-Xylene	EPA TO-15
Styrene	EPA TO-15
Toluene	EPA TO-15
Total Xylenes	EPA TO-15
Purgeable Halocarbons	
1,1,1-Trichloroethane	EPA TO-15
1,1,2,2-Tetrachloroethane	EPA TO-15
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA TO-15
1,1,2-Trichloroethane	EPA TO-15
1,1-Dichloroethane	EPA TO-15
1,1-Dichloroethene	EPA TO-15
1,2-Dibromoethane	EPA TO-15

Serial No.: 61079

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. KRISTINE A. DUSABLON
EUROFINS TESTAMERICA BURLINGTON
30 COMMUNITY DRIVE SUITE #11
SOUTH BURLINGTON, VT 05403

NY Lab Id No: 10391

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:

Purgeable Halocarbons

1,2-Dichloroethane	EPA TO-15
1,2-Dichloropropane	EPA TO-15
3-Chloropropene (Allyl chloride)	EPA TO-15
Bromodichloromethane	EPA TO-15
Bromoform	EPA TO-15
Bromomethane	EPA TO-15
Carbon tetrachloride	EPA TO-15
Chloroethane	EPA TO-15
Chloroform	EPA TO-15
Chloromethane	EPA TO-15
cis-1,2-Dichloroethene	EPA TO-15
cis-1,3-Dichloropropene	EPA TO-15
Dibromochloromethane	EPA TO-15
Dichlorodifluoromethane	EPA TO-15
Methylene chloride	EPA TO-15
Tetrachloroethene	EPA TO-15
trans-1,2-Dichloroethene	EPA TO-15
trans-1,3-Dichloropropene	EPA TO-15
Trichloroethene	EPA TO-15
Trichlorofluoromethane	EPA TO-15
Vinyl bromide	EPA TO-15
Vinyl chloride	EPA TO-15

Volatile Chlorinated Organics

Benzyl chloride	EPA TO-15

Volatile Organics

1,2-Dichlorotetrafluoroethane	EPA TO-15
1,3-Butadiene	EPA TO-15
1,4-Dioxane	EPA TO-15
2,2,4-Trimethylpentane	EPA TO-15
2-Butanone (Methylethyl ketone)	EPA TO-15
4-Methyl-2-Pentanone	EPA TO-15
Acetone	EPA TO-15
Carbon Disulfide	EPA TO-15
Cyclohexane	EPA TO-15
Hexane	EPA TO-15
Isopropanol	EPA TO-15
Methyl tert-butyl ether	EPA TO-15
n-Heptane	EPA TO-15
tert-butyl alcohol	EPA TO-15

Serial No.: 61079

ATTACHMENT 2

INTEGRAL DATA VALIDATION PERSONNEL RESUMES

Marcia Greenblatt, Ph.D., P.E.
Principal

Education and Credentials

Ph.D., Water Resources Engineering, University of California, Berkeley, 1997

M.S., Water Resources Engineering, University of California, Berkeley, 1993

B.S., Forestry, University of Massachusetts, Amherst, 1989

Professional Engineer, Massachusetts (License No. 48975), New York (License No. 100156)

Continuing Education and Training

Facilitation and Mediation of Public and Environmental Conflicts, Collaborative Decision Resources (2017)

The Transport of Sediment and Contaminants in Surface Waters, a short course taught by Dr. Wilbert Lick of University of California, Santa Barbara

Professional Profile

Dr. Marcia Greenblatt is a water resources engineer with 22 years of specialized experience in hydrodynamic, water quality, and sediment investigations. She has extensive involvement in CERCLA remedial investigations and feasibility studies (RI/FSs) at large sediment sites. Her responsibilities have included management of a major feasibility study and design of sediment, water column, and bathymetric data collection programs and field sampling plans. In addition, she has integrated data analyses within a geographic information system (GIS) to support site conceptualization, characterize and evaluate fate and transport processes, identify data gaps, parameterize and apply numerical models, and evaluate remedial alternatives. Dr. Greenblatt has designed and performed several modeling studies, applying both simple and complex numerical models, to predict hydrodynamic flows, sediment erosion, transport and deposition, chemical fate and transport, and water quality. She has performed numerous modeling studies for mixing zone evaluations to support NPDES renewals as well as operational evaluations, with several of these studies focused on thermal discharges. Dr. Greenblatt has served as both a testifying and consulting technical expert on water quality and sediment allocation matters.

Relevant Experience

Sediment Investigations

Lower Passaic River RI/FS, New Jersey—Project manager of the feasibility study, focused on evaluation of a source control interim remedy in the upper 9 miles of the 17-mile study area. If implemented, the interim remedy would be part of an adaptive management program. Participated in all aspects of this Lower Passaic River remediation project, including developing and managing sediment and water column field investigations; interpreting and analyzing data; and combining multiple lines of evidence (bathymetry, grain size, channel slope, and radiochemistry data) to support system understanding. Works closely with the project coordinators and project consulting team supporting project strategy development and technical review of project documents.

Yosemite Slough Pre-Remedial Design Investigation, San Francisco, California—Led a numerical modeling study to support an EPA

285 Century Place
Suite 190
Louisville, CO 80027

720.465.3347
mgreenblatt@integral-corp.com

non-time-critical removal action of lead- and PCB-contaminated sediments at the Yosemite Slough Superfund site. A hydrodynamic and sediment transport model was developed to assess cap stability to support engineering design. A field study was performed to collect site-specific data to set up and calibrate the models, and model predictions of bed shear stress and residual transport during tidal and storm conditions will be used in future design of capping and dredging remedy.

Big River Sediment Feasibility Study, Missouri—Served as project manager for feasibility study of a 50-mile reach of a mining-impacted river. The project included collecting sediment, soil, porewater, and tissue data to support a supplemental remedial investigation; developing a site conceptual model; updating the ecological risk assessment; performing a human health risk assessment; and identifying and evaluating remedial options. Developed and directed field investigations to support development of site-specific preliminary remediation goals to support evaluation of potential remedial options.

Sparrows Point, Baltimore, Maryland—Served as project manager of the investigation of potential offsite impacts of a steel mill to the surrounding harbor. Provided strategic support during scope development negotiations with state and federal agencies. Developed investigation approach, which included data collection, surface water modeling, and risk assessments.

Evaluation of Sediment Fate and Transport, Michigan—Technical expert supporting litigation on allocation evaluation of contaminated river sediments. Evaluated available data and previous court documents to develop a conceptual model of historical and ongoing sediment and contaminant transport at a Superfund site.

Engineering Evaluation and Cost Analysis (EE/CA) at a Former Chemical Manufacturing Facility, Portland, Oregon—Supported the EE/CA for sediment remediation at the site in Portland Harbor, including evaluating potential recontamination of remedial alternatives and application of a cap model to support development of conceptual design parameters and cost estimates.

Confidential Site, Washington—Provided senior planning and review for an analysis of the contributions of urban stormwater runoff to dioxin contamination in harbor sediments. Supported development and review of an analytical, GIS-based model using the revised universal soil loss equation and measured dioxin concentrations in municipal and regional soils to estimate the potential dioxin load associated with runoff and the associated liability to the municipality.

Dredge Disposal Monitoring, Providence Harbor, Rhode Island—Evaluated monitoring data collected during maintenance dredging. Developed confined aquatic disposal (CAD) cells and dredging disposal into the CAD cells at the Rhode Island Sound Disposal Site, as directed by the Providence River and Harbor Maintenance Dredging Project Water Quality Certification. Compared dredge material plume modeling results performed to support the environmental impact statement with monitoring results and recommended potential improvements for development of future dredged material monitoring plans.

Thermal Modeling and Evaluations

Thermal Discharge Evaluation, Vermont—Provided expert testimony in oral deposition, written testimony, and public hearings on the potential impact of thermal discharge from a nuclear power plant to receiving waters. Evaluated flow and temperature data and a hydrodynamic and thermal transport model to assess completeness of impact characterization.

Thermal Discharge Permit Evaluation and Litigation Support, Confidential Site—Evaluated existing NPDES thermal discharge permit and supporting modeling and data to assess validity and achievement of permit limits. Wrote expert opinion report.

Thermal Discharge Study, Kennedy Center for Performing Arts, Washington, DC—Designed a field program to characterize the thermal discharge into the Potomac River. The study was performed as part of NPDES compliance, and required that the Kennedy Center provide a temporal and spatial characterization of the thermal plume.

Thermal Discharge Study, Ohio—Designed a field program and modeling study as part of a thermal discharge study evaluating the refinery discharge plume to support an NPDES permit renewal. The field program includes long-term deployment of moorings and several boat-based surveys to delineate the thermal plume under a range of seasonal conditions. The modeling program includes both CORMIX (for nearfield analysis) and EFDC (farfield analysis) to predict the extent of the thermal plume under existing conditions not observed during the field study as well as future loading conditions. Developed the work plan, which included negotiation with and approval from Ohio EPA.

Thermal Discharge Analysis, Michigan—Evaluated the potential impact of increased thermal blowdown from the addition of a new unit at an existing nuclear power facility. Served as senior reviewer of the CORMIX model, which was used to predict the thermal mixing zone under typical and extreme monthly conditions, as well as rare events. The thermal discharge analysis also included data collection and review and statistical analysis of flow conditions. Presented the results on two occasions to the Nuclear Regulatory Commission.

Thermal Study of a Cooling Reservoir, Texas—Provided senior modeling oversight for the development of a 3-dimensional hydrodynamic and thermal model. Set up and applied the model to evaluate the potential impact of increased thermal load and reservoir reconfiguration on reservoir water temperatures. Project included characterization of reservoir temperatures and stratification, and calibration of the EFDC model to the observed temperatures. Ran the model to simulate a potential worst-case meteorological and loading condition to characterize temperatures across the reservoir.

Three-Dimensional Hydrodynamic and Thermal Modeling Study, Southeastern United States—Served as project manager and senior model reviewer, overseeing the development and application of the EFDC numerical model, which was used to evaluate the effect of moving a thermal discharge location on cooling water intake temperatures in a tidal estuary. The proposed channel bypass would move the heated discharge approximately 2 miles closer to the intake location. The model

was developed as a submodel to a regional model and calibrated to available water surface, salinity, and temperature measurements. Several months of plant operation were simulated, and predicted intake temperatures with and without the proposed bypass were compared to support the decision-making process surrounding the construction of the bypass channel.

Hydrodynamic, Sediment, and Water Quality Studies

Florida v. Georgia No. 142, Original—Testified as an expert witness in the U.S. Supreme Court in an interstate water rights case. Performed hydrodynamic modeling to evaluate water quality impacts due to upstream water use.

Barr Lake and Milton Reservoir Nutrient Total Maximum Daily Load (TMDL) Water Quality Model, Colorado—Served as project manager for the expansion (including new input data) and recalibration of a water quality model to support assessment of TMDL alternatives for the Barr Lake and Milton Reservoir Watershed Association Technical Committee. The linked watershed/in-lake model simulated flows and nutrients in a complex system with an extensive canal system, numerous irrigation inputs and withdrawals, and two water supply reservoirs. The EPA models SWAT and WASP were applied to ultimately predict present and potential future in-lake nutrient systems in a severely eutrophic system.

Sedimentation and Water Quality Modeling Study, Alaska—Led a study to evaluate the potential sedimentation and turbidity impacts due to proposed construction of a trans-Alaska liquefied natural gas pipeline. Developed a 3-dimensional hydrodynamic and sediment transport model using Delft-3D to predict water column turbidity concentrations and sedimentation based on proposed construction activities. Models were set up in a tidal bay and in multiple freshwater streams to characterize the range of potential impact. Submitted modeling reports as part of the Federal Energy Regulatory Commission application to support the environmental report.

Water Quality and Fish Tissue Field Study, Confidential Client—Designed and implemented a field program to evaluate recent water quality and tissue concentrations for emerging contaminants. The program was designed to characterize fluxes, identify potential upland sources, and compare to previous studies. Program was designed and implemented in an expedited timeframe to support ongoing negotiations.

Three-Dimensional Hydrodynamic and Sediment Transport Modeling Study, Gulf of Mexico—Set up and applied the EPA-supported EFDC model to evaluate potential water quality and benthic habitat impacts associated with proposed construction of a pipeline from Tampa Bay through the Gulf of Mexico. Applied the model to predict tidally varying currents and sediment transport and deposition potentially resulting from pipeline construction activities. Modeling activities included application of the U.S. Army Corps of Engineers ADDAMS models DREDGE and STFATE to estimate sediment resuspension rates from dredging and sidecasting operations, and mapping model-predicted suspended sediment concentrations and sediment deposition extents and depths over sensitive habitat areas.

Dredge Disposal Evaluation, Savannah Harbor, Georgia—Applied the ADDAMS STFATE model to evaluate the fate and transport of disposed dredge material in Savannah Harbor. Used field data to set up the model. Conducted model simulations to estimate sediment transport and water column concentration. Model results were interpreted to determine whether relevant water quality standards were achieved within the disposal area.

Industrial Effluent Modeling in Tidal Estuary, Nova Scotia, Canada—Modeled nearfield and farfield dilution and mixing of an industrial effluent discharge into a tidal estuary to support the identification of alternate locations for relocating an existing outfall. Used the 3-dimensional hydrodynamic and water quality model HEM3D to simulate a conservative tracer and predict the extent of the visible plume. A number of locations and discharge scenarios (continuous, hold-and-release) were investigated. Calibrated the farfield model by using measurements taken during a synoptic field survey specially designed to support the modeling task. Conducted nearfield mixing modeling to evaluate alternative diffuser designs.

Nonpoint Source Modeling for Nutrient TMDL Study, Massachusetts—Modeled hydrology, water quality, and biology in a eutrophic river system in support of the TMDL process. Project included several rounds of field data collection and analysis, model selection, setup, and development. Set up an application of the HSPF watershed and instream numerical model to simulate point and nonpoint source flow and nutrient loads into the river, as well as instream nutrient and biomass interactions. Set up the model based on GIS data, and calibrated and validated the model using several sets of field data. Once calibrated, applied the model to assess several alternative management scenarios.

Oil Spill Modeling, Brazil—Used available bathymetry and shoreline data to set up and apply a hydrodynamic and oil spill model to assess the impact of a potential oil spill in support of a permit application. The dynamic model was calibrated to measured current and water surface elevation data collected for the study. The predicted flow fields were imported into a particle tracking oil spill model. The oil spill model was applied for several dominant wind conditions and several types (varying in location, duration, and volume) of oil spills. The areal extent and location of the spill were tracked for several days. The extent of coastline potentially impacted and the time until impact were assessed for each modeled scenario.

Hurricane Protection Office, New Orleans, Louisiana—To support the design-build of a 9,000-ft hurricane barrier in coastal Louisiana, developed a numerical model to evaluate channel velocities during construction and operation of the barrier sector gates. Designed and managed the field program implemented to characterize the flows and circulation patterns in the study area. An RMA-2 model was set up, calibrated, and validated to a large set of available field data throughout the model domain. The calibrated model was applied to predict velocities for a set of design scenarios, and the model results were used to support the design team in construction and operations planning.

Temperature TMDL Study, Vermont—Developed a numerical model (SNTEMP) to simulate water temperature in a river in support of the TMDL process. Field data were used to calibrate and

validate the model. The model was applied to assess the effects of alternative management scenarios on instream water temperature. Model results were compared to the requirements of native and/or desired species to determine the optimum alternatives for adequate habitat.

Mixing Zone Studies and National Pollutant Discharge Elimination System Permitting

Discharge Permit Renewal Evaluation, American Samoa—Provided consulting expertise and strategic direction to support renewal of a NPDES permit to discharge nutrients into a harbor. Work included review of existing data and mixing zone modeling, development of CORMIX initial mixing model and DELFT3D far-field numerical model, and submittal of an expert report to support comments on draft permit.

Anti-Degradation Evaluation, Ohio—Evaluated the impact of cessation of a groundwater pump and treat system on downstream water quality. Applied state guidance to model potential future water quality for evaluation of anti-degradation potential.

Thermal Discharge Permit Evaluation, Confidential Site—Evaluated existing NPDES thermal discharge permit and supporting modeling and data to assess validity and achievement of permit limits. Wrote expert opinion report.

Mixing Zone Analysis, Gilbert Generating Station, New Jersey—Applied CORMIX expert mixing zone model to determine the extent of the mixing zone induced by a cooling water discharge. Compared results with applicable water quality standards.

Water Discharge Permitting, Wythe, North Carolina—Modeled the mixing zone of a cooling tower discharge into an adjacent river for several constituents of interest. Evaluated alternative diffuser designs to determine the best design and location for maximum mixing.

Water Supply and Discharge Design and Permitting, Rensselaer, New York—Evaluated siting intake and discharge locations to avoid recirculation on the tidally reversing river for a proposed newsprint facility on the Hudson River. The potential to entrain fish larvae at the intake was evaluated on a seasonal basis for the vulnerable life stages of each species. The mixing zone of the effluent was evaluated for various times during the tidal cycle under a range of freshwater inflow conditions. The optimal diffuser design was developed to meet the mixing requirements and the physical constraints within the river (including rapidly changing bathymetry and the proximity to a shipping channel).

Oceanographic Studies

Disposal Area Monitoring System (DAMOS), New England—Served as program manager for the DAMOS program for monitoring of disposed sediment and benthic habitat recovery at dredged material disposal sites in coastal New England and New York. Budgeted tasks, planned resources and surveys, and coordinated subcontractors. Oversaw monitoring cruises that typically included bathymetric, side-scan, and sediment-profile imaging (SPI) surveys after the disposal season. Supported the development of bathymetric maps and depth-difference maps, interpretation of SPI

results, and comparison of disposal area sediment data with reference and historical data. Wrote and reviewed technical reports included as DAMOS contributions and distributed to the public. Coordinated the 10th and 11th DAMOS symposia, where recent DAMOS work and new technological advances were presented to government representatives (state and federal) as well as the interested public.

Physical Oceanographic Evaluation, Long Island Sound, New York—As part of a major EPA-required environmental impact study to assess potential impacts associated with dredged material disposal and evaluate alternative candidate disposal sites in Long Island Sound, performed extensive oceanographic data analysis, including time series analysis and spectral analysis of water velocity and wave data, and developed a graphical presentation of all historical and recent data. Specific tasks included an intensive review of historical physical oceanographic data, creation and distribution of a GIS-based database of all physical oceanographic data on Long Island Sound, collaboration and meetings with the research community in the sound to reach consensus on required future hydrodynamic data collection, and selection of optimal disposal site locations in terms of hydrodynamic and sediment transport objectives.

Publications / Presentations

Greenblatt, M., W. Locke, and R. Law. 2019. Adaptive management: A practical approach to remediation of the Lower Passaic River. Platform presentation at Battelle 10th International Conference on the Remediation and Management of Contaminated Sediments, New Orleans, LA. February 11–14.

Greenblatt, M., and J. Connolly. 2015. Lower Passaic River (RM 10.9) early action: Evaluation of remedial design data and implications for river-wide remedy development. Battelle 8th International Conference on Remediation of Contaminated Sediments, New Orleans, LA. January 12–15.

Thorvaldsen, A., G. Dang, P. Israelsson, J. Connolly, P. Oates, and M. Greenblatt. 2015. A contaminant mapping methodology for remedial alternatives assessment on the Lower Passaic River. Battelle 8th International Conference on Remediation of Contaminated Sediments, New Orleans, LA. January 12–15.

Greenblatt, M., M. Barbara, J. Connolly, and R. Law. 2011. Lower Passaic River conceptual site model—Integration of multiple lines of evidence. Battelle 6th International Conference on Remediation of Contaminated Sediments, New Orleans, LA. February 8.

Barbara, M., M. Greenblatt, and J. Connolly. 2010. Sediment stability in the Lower Passaic River—Integration of multiple lines of evidence. Fourth Passaic River Symposium, Montclair, NJ. June 22.

Connolly, J., M. Greenblatt, L. Postma, H. Winterwerp, R. Canizares, and R. Law. 2009. Interpretation of spatial patterns of contaminants in the Lower Passaic River. SETAC North America 30th Annual Meeting, New Orleans, LA. November 19–23.

Law, R., J. McNally, R. Canizares, and M. Greenblatt. 2009. Evaluation of sediment stability in the Lower Passaic River using the weight-of-evidence approach. SETAC North America 30th Annual Meeting, New Orleans, LA. November 19–23.

Greenblatt, M., S. Wolf, and T. Fredette. 2009. Providence River and Harbor maintenance dredging project—summary and lessons learned. Western Dredging Association XXIX Conference and 40th Texas A&M Dredging Seminar, Tempe, AZ. June 14–17.

Gerath, M., and M. Greenblatt. 2008. Evaluation of hydrologic disturbance frequency and duration in western ephemeral streams. In: Relevance of ambient water quality criteria for ephemeral streams and effluent-dependent watercourses of the arid western United States. R.W. Gensemer, R.D. Meyerhoff, K.J. Ramage, and E.F. Curley (Eds.). Pensacola, FL. Society of Environmental Toxicology and Chemistry. 268 pp.

Ruffle, B., M. Greenblatt, and D. Reid-Green. 2007. Application of geostatistics and risk assessment to property divestitures. University of Massachusetts Annual Conference on Soils, Sediments, Water, and Energy, Amherst, MA. October 15–18.

Wolf, S., M. Greenblatt, and T. Fredette. 2006. Stability and recovery of capped in-channel CAD cells—Boston Harbor, MA. Western Dredging Association XXVI Conference and 38th Texas A&M Dredging Seminar, San Diego, CA. June 25–27.

Greenblatt, M. 2003. Modeling aquatic biology: A TMDL challenge. 27th Annual Meeting of the New England Association of Environmental Biologists, Wachusett, MA. March 26–28.

Greenblatt, M., K. Hickey, and K. Heim. 2001. Riverine nutrient TMDL allocation: overview of the field program and modeling application. In: Proc. of the 2nd ASCE Wetlands Engineering and River Restoration Conference. Reno, NV. American Society of Civil Engineers.

Morin, I., K. Hickey, M. Greenblatt, and G. Gong. 2000. Using GIS as an interface for 3D hydrodynamic modeling. Estuarine and coastal modeling. In: Proc. of the 6th International Conference, New Orleans, LA. November 1999.

Hickey, K., I. Morin, M. Greenblatt, and G. Gong. 2000. 3D hydrodynamics of an estuary in Nova Scotia. Estuarine and coastal modeling. Proc. of the 6th International Conference, New Orleans, LA. November 1999.

Gilman, J, J. San Antonio, M. Greenblatt, and S. Emmons. 2010. Application of RMA2 for design and construction of the inner harbor navigation canal hurricane surge barrier. In: 83rd Annual Water Environment Federation Technical Exhibition and Conference; WEFTEC. New Orleans, LA.

Greenblatt, M.S., and R.J. Sobey. 1998. Near surface flow and transport in tidal wetland marsh plains. In: Proc. of the ASCE Wetlands Engineering and River Restoration Conference. Denver, CO. American Society of Civil Engineers.

Greenblatt, M.S., and R.J. Sobey. 1999. Subsurface flow and transport in tidal wetlands: Marsh plain equations. *J. Engr. Mech*. 125(8):971–974.

Greenblatt, M.S. 1997. Surface water–groundwater interactions in a tidal wetland marsh plain. Ph.D. thesis. University of California, Berkeley, Berkeley, CA.

Greenblatt, M.S., and R.J. Sobey. 1997. Saturated/unsaturated flow and salinity transport using method of lines. pp. 907–912. In: Environmental and Coastal Hydraulics: Protecting the Aquatic Habitat. Water for a Changing Global Community: Proc. of the XXVII Congress. International Association for Hydraulic Research, San Francisco, CA.

Greenblatt, M.S., and R.J. Sobey. 1997. Subsurface flow and salinity response patterns in a tidal wetland marsh plain. In: Tidal Wetland Management: Integrated Ecological and Physical Processes. Proc. of the XXVII Congress. International Association for Hydraulic Research, San Francisco, CA. Short Course Notes.

Jeffrey E. Marsh, E.I.T.
Consultant

Education and Credentials

B.S., Chemical Engineering, Clarkson University, Potsdam, New York, 2003

New York Intern Engineer (Engineer in Training) (License No. 083740)

Continuing Education and Training

Hazardous Waste Operations and Emergency Response 40-Hour Certification (2003; refreshers annually)

OSHA Confined Space Training (2015)

First Aid and CPR Certified (2018)

Professional Affiliations

Member of Air and Waste Management Association

Achievements and Awards

American Council of Engineering Companies 2018 Diamond Award

Trenchless Technology 2016 Rehabilitation Project of the Year Honorable Mention

Western Dredging Association 2014 Health and Safety Excellence Award

Professional Profile

Mr. Jeffrey Marsh has 16 years of experience in the fields of chemical and environmental engineering. He is currently responsible for delegating technical responsibilities to engineers, designers, and drafters on complex and diverse projects. He has an active role in developing project budgets and schedules, preparing reports, managing projects, and maintaining a close relationship with clients, regulatory agencies, and other stakeholders throughout the development of a project.

His experience includes monitoring and management of remedial investigations; design, construction, startup, operation, monitoring, and troubleshooting of remedial systems; development of treatment solutions to remedy contaminated sites; and preparation of feasibility studies, as well as air and water permitting. Mr. Marsh is also experienced in the design, implementation, and troubleshooting of computer control systems. He has experience preparing cost estimates and engineering input for feasibility studies at CERCLA sites and with large-scale groundwater remediation system design, construction, and operation and with habitat restoration within floodplain environments.

Relevant Experience

Remedial Investigation, Design, Construction, and Operation

Groundwater Collection, Conveyance, and Treatment System Design and Construction, Onondaga Lake Superfund Site, Geddes, New York—Managed the design of an interim remedial measure (IRM) and oversaw construction for groundwater collection systems, low permeability lining systems, groundwater pumping systems, and groundwater treatment plant. Project included design and construction of more than 7,000 ft of groundwater collection trench, a treatment plant designed to reduce groundwater pH for direct discharge to publicly owned treatment works, and upgrades to an existing pumping station. Groundwater contamination included primarily metals, volatile organic compounds (VOCs), and semivolatile organic compounds (SVOCs), with a pH of up to 12 and high scalability.

1001 6th Avenue
11th Floor
New York, NY 10018

315.651.2020
jmarsh@integral-corp.com

Groundwater Collection System and Storm Sewer Rehabilitation Design and Construction, Onondaga Lake Superfund Site, Geddes, New York—Managed development of IRM design for rehabilitation of existing interstate highway storm sewers, groundwater collection systems, low permeability lining systems, and habitat restoration. Project included mitigation of contaminated seepage affecting a berm proximate to a public highway via low-permeable lining systems and a groundwater collection system, and reduction of existing highway storm sewer infiltration via cured-in-place-pipe. Contamination included high concentrations of VOCs, primarily benzene, chlorobenzene, and dichlorobenzene.

Groundwater Collection System and Storm Sewer Rehabilitation Pre-design Investigation and Design, Onondaga Lake Superfund Site, Geddes, New York—Managed development and implementation of a pre-design investigation program, including topographic survey, geotechnical borings, monitoring well installation, *in situ* hydraulic conductivity testing, groundwater and surface water sampling and level monitoring, culvert inspection and sampling, vegetation survey, geotechnical sample testing, and hydrogeologic investigation. Subsequently, managed development of IRM design for groundwater and seep collection, treatment and conveyance, low permeability lining systems, culvert and manhole rehabilitation, and habitat restoration. Groundwater contamination included primarily metals, VOCs, and SVOCs.

Groundwater Pump Station Pre-design Investigation and Design, Onondaga Lake Superfund Site, Geddes, New York—Managed pre-design investigation and development of IRM design and provided construction oversight for multiple groundwater pump stations. Pump stations were installed to convey collected groundwater to a central treatment facility located more than 2 miles away. Groundwater contamination included primarily metals, VOCs, and SVOCs, with a pH of up to 12 and high scalability.

Stormwater Lift Station and Storm Sewer Rehabilitation Design and Construction, Onondaga Lake Superfund Site, Geddes, New York—Performed and provided oversight for day-to-day design tasks, including development and review of specifications, drawings, and reports, associated with an IRM at a former industrial site. Provided oversight and reviewed submittals during construction. IRM included lining and rehabilitation of existing storm sewers impacted by mercury and VOCs, and installation of a stormwater lift station designed to bypass a 150-year-old, 40-ft-deep, hand-laid, brick storm sewer pipe experiencing heavy infiltration. Peak flows were approximately 5,000 gallons per minute (gpm) with an average flow of approximately 30 gpm.

Groundwater Collection and Conveyance, and Brook Sediment Excavation and Restoration Design and Construction, Onondaga Lake Superfund Site, Geddes, New York—Performed and provided oversight for day-to-day design tasks, including development and review of specifications, drawings, and reports, associated with an IRM at a former industrial site. Provided oversight and reviewed submittals during construction. IRM included low permeability liner systems, groundwater collection and conveyance systems, culvert rehabilitation, sediment removal, and wetland, stream, and habitat restoration. Project included construction of two groundwater pump stations, installation of approximately 4,000 ft of groundwater collection and conveyance, rehabilitation of a former railroad bridge eligible for listing on the National Register of Historic

Places, and sediment removal within a Class C stream. Project included a large permitting effort, because the site was owned by 13 different public and private property owners, including the New York State Department of Transportation and CSX, and was transected by more than 15 different utility rights of way. Groundwater contaminants included primarily metals, VOCs, and SVOCs, with a pH of up to 12, high scalability, and a free product (nonaqueous-phase liquid, NAPL) plume.

Geothermal Testing and Design, New Haven, Connecticut—Designed geothermal pilot testing procedure for standing column geothermal well system. Project included the first use of pilot testing for standing column wells to support geothermal design in the United States. Tasks included full-scale pilot testing followed by design and specification of geothermal system.

Geothermal Design for International Conference Center, New Haven, Connecticut—Designed standing column geothermal well system. Tasks included design and specification of geothermal system developed to achieve high standard of noise reduction and aesthetic improvements for international conference center.

Geothermal Design and Optimization, New Haven, Connecticut—Designed standing column geothermal well system. Tasks included design and specification of geothermal system for conference centers as well as optimization studies to investigate the most cost-effective combination of geothermal and central plant systems.

Dual-Phase Extraction System, Rochester, New York—Performed dual-phase, vacuum-enhanced, pumping pilot test for the removal of petroleum hydrocarbons in a residential neighborhood affected by leaking tanks at a nearby gasoline station. Used pilot test results to design a full-scale remedy consisting of dual-phase extraction with subsequent treatment of water and vapor streams via oxidation and air stripping. Also, managed and coordinated day-to-day operations and maintenance of full-scale remedy.

Pump and Treat System, Defiance, Ohio—Designed a pump and treat system for the removal of PCBs from groundwater at a foundry. The system use ultrafiltration to remove PCBs sorbed to solids. Prepared design drawings and assisted with system startup.

Soil Vapor Extraction/Bioremediation System, North Hollywood, California—Managed source removal activities for an active soil vapor extraction system for the removal of petroleum hydrocarbons from the subsurface. Work included monitoring and evaluating operating conditions to recommend system adjustments to optimize operation. Also worked as project engineer for the design of a hybrid remediation system for the downgradient methyl *tert*-butyl ether plume. The system combines *ex situ* treatment with biologically seeded carbon beds and oxygenation of the groundwater for *in situ* enhanced biological degradation. Work included an IRM design and work plan, preliminary and final design of a full-scale remedy, and system startup and monitoring. Teamed with regulators to become the first site ever in the state of California to reinject treated groundwater back into a drinking water aquifer.

Dual-Phase Extraction System, Lodi, New Jersey—Designed a dual-phase extraction system for the removal of chlorinated solvents from groundwater. The system uses a catalytic oxidizer, scrubber, carbon, resin, and air stripper to remove the contaminants of concern. Prepared design drawings and assisted with system startup and long-term operations. Also coordinated short-term operations and maintenance, such as water and air sampling, well-field data collection, and control loop tuning.

Dual-Phase Extraction System, Boston, Massachusetts—Designed a dual-phase extraction system for the removal of chlorinated solvents and petroleum hydrocarbons from groundwater. The system uses a catalytic oxidizer and air stripper to remove the contaminants of concern. Prepared design drawings, equipment specifications, and procurement quotes.

Vacuum-Enhanced Pumping/Dual-Phase Extraction System at Chemical Plant, Resende, Brazil—Designed a dual-phase extraction and vacuum-enhanced pumping system for the removal of multiple high-concentration contaminants from groundwater at an active chemical plant in Brazil. Prepared design drawings and assisted with system startup. Coordinated short-term operations and maintenance with plant personnel who had very limited understanding of the technologies employed, such as water and air sampling, well-field data collection, system optimization, startup, and control loop tuning.

Dual-Phase Extraction System, Argentina—Designed a dual-phase extraction system for the removal of multiple high-concentration contaminants from groundwater at a site in Argentina. Prepared design drawings and assisted with system startup. Coordinated short-term operations and maintenance with plant personnel who had very limited understanding of the technologies employed, such as water and air sampling, well-field data collection, system optimization, startup, and control loop tuning.

Brownfield Redevelopment Site, Tarrytown, New York—Assisted in the remediation design of a former manufactured gas plant site being redeveloped for use in a residential capacity. Performed and oversaw confirmation testing, with oversight from the New York State Department of Environmental Conservation, to ensure that no vapor intrusion into residential living spaces was occurring.

FERC Permitting Project, Florida-Bahamas—Assisted in the permitting process for a $550 million installation of a 54-mile liquefied natural gas line from Ocean Cay (near Bimini, Bahamas) to Broward County, Florida. Evaluated impacts to critical habitats of any endangered or at-risk species.

Landfill Closure, Storrs, Connecticut—Designed a leachate collection and pumping system at a former landfill. Work included design of pumping and control systems, review of contractor submittals, assistance with startup and troubleshooting, and confirmation of proper operation.

Soil Vapor Intrusion System, Wampsville, New York—Designed a sub-slab depressurization system to prevent vapor intrusion into an active industrial facility. Oversaw construction activities and subsequent confirmation sampling and testing.

Blasted Bedrock Collection System, Rochester, New York—Designed a blasted bedrock trench and associated groundwater pumping system. Tasks included system design, programmable logic controller (PLC) design, and system startup.

Pump and Treat System at Automotive Plant and Landfill, Farmington, New Hampshire—Assisted in the design of a pump and treat system at an automotive plant and inactive landfill. Tasks included assistance with design decisions, preparation and review of contract drawings, and design of PLC system.

Pump and Treat System at Aerospace Facility, Huntington Beach, California—Designed a pump and treat system to remediate a large groundwater plume with multiple contaminants at an active aerospace facility. Tasks included assistance with design decisions, preparation and review of contract drawings, and design of electrical and PLC system.

Design and Permitting for Research and Development Facility, Canandaigua, New York—Assisted with design and permitting of a multi-purpose research and development facility. Tasks included evaluating materials compatibility for conveyance of multiple highly corrosive, acidic, basic, and explosive chemicals; designing treatment trains for multiple waste streams; and working with permitting lead to ensure a seamless permitting process.

Bottled Water Permitting Application, Ecuador—Provided engineering and permitting support to a large international bottling company seeking a permit to sell bottled water in New York State. Reviewed existing bottling operations to provide engineering recommendations to meet New York State Health Department and U.S. Food and Drug Administration regulations, and reviewed analytical data to evaluate compliance with applicable regulations.

Remediation System Monitoring and Optimization

Groundwater Collection and Habitat Restoration Performance Verification and Monitoring, Onondaga Lake Superfund Site, Geddes, New York—Managed implementation of performance verification and monitoring for several existing IRMs, including groundwater collection and conveyance systems, wetland and habitat restorations, and low permeable lining systems. Performance verification and monitoring activities included collection and analysis of operational data, verification that systems were meeting regulatory compliance requirements, media sampling, development and implementation of corrective actions, and annual reporting.

Dense Nonaqueous-Phase Liquid Recovery System Optimization, Onondaga Lake Superfund Site, Geddes, New York—Managed development, design, and implementation of a performance optimization strategy for an existing dense NAPL recovery system.

Nonaqueous-Phase Liquid Recovery System, Los Angeles, California—Performed operation, maintenance, and monitoring activities for a NAPL recovery system consisting of more than

200 recovery wells, in addition to a groundwater and air treatment system, to evaluate and optimize system operation. Tasks included continuously operating the remediation systems, assisting technicians with system adjustments to improve recovery, and updating and improving the computer control system.

Geothermal Performance Improvement Evaluations, Cambridge, Massachusetts—Performed evaluations to improve performance of existing geothermal well system, including developing recommendations for increased reliability through use of changes in control systems.

Pump and Treat/Soil Vapor Extraction System, Morristown, Tennessee—Oversaw quarterly groundwater sampling events and used data to determine effectiveness of soil vapor extraction and pump and treat systems. Managed site operations and maintenance contractor to maintain maximum system uptime. Monitored system operation, and recommended and designed system upgrades.

Pump and Treat System, Rochester, New York—Coordinated operations, maintenance, and sampling activities for a pump and treat system at a former industrial site. Recommended and designed system upgrades to increase and maintain uptime requirements. Used site groundwater and system data to recommend operational changes.

Feasibility Studies

Focused Feasibility Evaluation, Hinkley, California—Provided highly detailed cost estimates for an array of remedy options related to cleanup of a chromium contaminated groundwater plume approximately 2 mi^2 in size. Tasks included developing modular cost estimates that could be applied to various remedy options, evaluating proposed remedy effectiveness at meeting project goals, and presenting the data in a manageable and meaningful form to various stakeholder groups.

Environmental Liabilities Portfolio Estimates, Various Locations—Assisted with the development of detailed cost estimates for the environmental liabilities portfolio of a large domestic automobile corporation. Worked with a multi-disciplinary engineering team to develop comprehensive estimates for multiple sites with a multitude of environmental contaminants. Estimates were used to develop a portfolio-wide liability reserve for use in corporate bankruptcy hearings.

Presentations / Posters

Marsh, J. 2017. Overcoming several site-specific challenges to remediate an urban brook and several tributaries. Ninth International Conference on Remediation and Management of Contaminated Sediments, New Orleans, LA.

Glenn Esler
Scientist

Education and Credentials

B.S., Geography, Portland State University, Portland, Oregon, 2008

A.S., Chemistry, Millersville University, Millersville, Pennsylvania, 1984

Continuing Education and Training

Sustainability Leadership Program Certificate, University of Oregon, Portland, Oregon (2013)

EPA Office of Emergency and Remedial Response, 40-Hour Health and Safety Course (2010)

Certified Laboratory Auditor Training and Credentialing Program, iNARTE (2009)

Naval Sea Systems Command Laboratory Quality and Accreditation Office Sampling and Laboratory Testing E-Learning Training (2009)

Radiometric Data Validation, American Radiochemistry Society (2009)

SDSFIE Web Online Training Course (2005)

Analysts Guide to NELAC Assessment Short Course, Advanced Systems, Inc. (2004)

Basics of Quality Improvement Short Course, University of Delaware (1996)

Environmental Data Quality Short Course, American Chemical Society (1992)

Professional Profile

Mr. Glenn Esler has more than 30 years of experience in the field of environmental chemistry, including 15 years in laboratory quality assurance and data quality management and 5 years as a GC/MS analyst. His technical specialties include design and implementation of laboratory quality management programs, laboratory and field audits, and data interpretation and assessment of compliance with regulatory requirements and project objectives. He has an in-depth working knowledge of EPA environmental analytical methods and EPA Contract Laboratory Program (CLP) national functional guidelines for data review. His experience includes environmental analysis, data verification and validation, preparation of quality assurance documentation, and coordination of subcontracting laboratories. He is also credentialed as a Certified Laboratory Auditor.

Relevant Experience

Quality Assurance and Quality Control

Airplane Manufacturer Superfund Site, Laboratory and Field Audits, Washington—Conducted onsite laboratory and field audits in support of remedial action and treatment systems related to groundwater contamination. Wrote final report that provided an assessment of the laboratory and field sampling team's performance and ability to provide high-quality, defensible data, and areas where improvements are required.

NOAA, Lower Duwamish River (LDR), Washington—Conducted research related to the Natural Resources Damage Assessment program for PAH allocation in LDR sediments. Research was based on PAH footprint maps, tax parcel information, data from EPA and Washington State Department of Ecology files, site histories, and other publicly available reports produced over the last several decades. Also used Google Earth and ESRI's ArcView to aid in allocation to multiple sites along the LDR.

Energy Distribution Company, Indiana—Assisted with work plan preparation, laboratory coordination, and data validation, data review, and data quality assessment on public sewer sediments and stormwater sampling at the site. The site was identified as a

319 SW Washington St.
Suite 1150
Portland, OR 97204

503.943.3617
gesler@integral-corp.com

potential source of PCBs to a public sewer system and river sediments associated with a National Priorities List site.

Railroad Transportation Laboratory Audits, Multiple Sites, United States—Conducted onsite laboratory audits and provided assistance in conjunction with the Laboratory Management Program. The program included establishment of a web site for distributing program information, development of a web-based project management tool to handle laboratory projects, documentation of laboratory procedures in an online and hard copy manual, solicitation and establishment of standardized pricing for laboratory work, and presentation of the program to railroad officials, laboratories, and consultants. Also audited laboratories analyzing NPDES samples on behalf of client; evaluated laboratory reports for completeness, verification of reporting limits, and laboratory standard operating procedures. Wrote final report that provided an assessment of the laboratory's performance and ability to provide high quality, defensible data, and areas where improvements were required.

Cleanup of Base Oil/Water Separators, Air Force Center for Environmental Excellence, Grissom Air Reserve Base, Indiana—Assisted with quality assurance project plan (QAPP) preparation and data quality objectives (DQOs) and performed data validation, data review, and data quality assessment in conjunction with site activities, which included sampling, analyzing, cleaning, collecting, removing, manifesting, and properly disposing of materials for nine oil/water separators in accordance with applicable state regulations.

Selfridge Air National Guard Base, Michigan—Assisted with QAPP preparation and formulation of DQOs for the collection of data to support the evaluation of the corrective action measures, site characterization, and determination of extent of contamination at a Michigan Air National Guard Base.

U.S. Department of the Navy, Naval Facilities Engineering Command Southwest, California—Assisted with the preparation of the pre-design sampling and analysis plan (SAP) and remedial action work plan for the remedial design and remedial action at IR Site 1. Also assisted with laboratory procurement of analytical services and procurement of third-party data validation services.

Groundwater Monitoring Program, Arizona—Assisted in the development of the site-wide quality assurance management plan and the QAPP for an EPA Superfund site. Contaminants of concern were volatile organic compounds (VOCs) and perchlorate. Activities included groundwater program planning and execution, groundwater sampling, quarterly and annual reporting, QA/QC, data validation, and project problem solving. Supported the project quality assurance manager by providing data validation, tracking quality control parameters, and handling laboratory data quality issues.

Partial Database Rebuild for a Sawmill Facility, Montana—Provided technical support for the partial reconstruction of the project database after discrepancies were found during quality

assurance activities. Review third-party data validation reports and updated associated electronic data deliverables as appropriate.

Emergency Response at Bulk Chemical Terminal, New Orleans, Louisiana—Assisted with data analyses and audit of the analytical laboratory charges for samples collected related to the emergency response and cleanup of a chemical spill caused by flooding of a bulk chemical terminal during Hurricane Isaac.

Engineering Evaluation and Cost Analysis for a Former Chemical Manufacturing Facility, Portland, Oregon—Revised project QAPP based on EPA comments on a sediment sampling work plan, which was prepared to collect data for pre-remedial design to address sediments adjacent to the site. Coordinated with analytical laboratories for methods, quality control criteria, standard operating procedures, quality assurance documentation, and costs for additional analyses. Researched and co-authored technical memorandum to EPA on the passive sampling effort to measure the freely dissolved porewater concentrations of DDT and its metabolites, polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs), and PCBs described in the porewater chemistry section of the work plan.

Laboratory Forensics Investigation, Oregon—Supported the verification of possible reporting anomalies initiated by a respected commercial laboratory. Performed preliminary data review activities, including review of laboratory documentation, quality control data, and selected instrument run data files followed by a more comprehensive review process for instrument run file outputs associated with reported data.

Forensics Investigation Municipal Wastewater Collection and Treatment Facility, Oregon—Supported a third party investigation of possible analysis procedural and data integrity issues associated with a municipal wastewater treatment plant. The project entailed a review of electronic data files, permit requirements, laboratory record books, and laboratory standard operating procedures, laboratory audits, and staff interviews. Included review of laboratory and corporate procedural guidance documents, instrument manuals, laboratory bench books, and discharge monitoring report data submitted in fulfillment of NPDES requirements. The technical evaluation included data verification by tracing records from sample analysis through reporting, evaluation of quality control data for compliance with laboratory control limits, visual evaluations of time series data and trends, and assessment of the impact of possible improper laboratory practices.

Litigation Support

Database Inventory in Support of Litigation, U.S. West Coast—Supported maintenance of database inventory, which included a summary of relevant information such as information types, sample material, geographic area, period of record, and source information for customized databases cataloging large numbers of publicly available data sets.

Biomonitoring Study Conducted in Support of Litigation, Missouri—Evaluated laboratory methodologies and data usability and prepared a report summarizing the data usability results associated with the collection of human serum from more than 500 participants.

Expert Testimony Report, Confidential Client—Performed research on the pervasiveness and persistence of organochlorine pesticide chemicals in environmental media and biota in support of expert testimony report.

Project Chemistry

Railyard Air Monitoring, Various Sites, Montana—Served as project chemist for semiannual air sampling program related to indoor air monitoring at several active railyards throughout Montana. Oversaw data validation effort using various air analytical methods, including EPA TO-15 and MADEP VPH. Reviewed data validation reports and associated electronic data deliverables.

Air National Guard, One Clean Program, Multiple Sites, North/Midwest Region—Served as project chemist and oversaw preparation of the QAPP, data validation, and data management for this accelerated turnaround project, which included field investigation activities to determine the presence of environmental contamination at identified areas of concern at 38 sites at 11 installations in the Air National Guard's North/Midwest Region. Oversaw the following: management of all analytical data using the EQuIS™ data management tool; Level III data validation consistent with the Environmental Restoration Program Air National Guard Investigation Guidance; creation of export templates from the database; generation of data tables for the Site Inspection Report; and the electronic data deliverables for the ESOH-MIS database.

Niblack Mining Corporation, Ketchikan, Alaska—Prepared a QAPP revision in support of routine monitoring of surface water and groundwater quality. Assisted in coordinating project logistics, sending sampling equipment to a remote location in Alaska, and subsequent delivery of samples to the analytical laboratory. Monitored laboratory's progress on sample analyses and reviewed and validated analytical results. Supported preparation of data quality reports summarizing analytical results.

Water Quality Monitoring for a Volcanogenic Massive Sulfide Mine Exploration Project, Alaska—Assisted with QAPP preparation in support of monitoring of surface water and groundwater quality. Assisted in coordinating project logistics, sending sampling equipment to a remote location in Alaska, and subsequent delivery of samples to the analytical laboratory. Monitored laboratories' progress on sample analyses and reviewed and validated analytical results.

Baseline Ecological Risk Assessment (BERA) for a Landfill Superfund Site, New Jersey—As the project's quality assurance chemist, assisted with QAPP preparation for analytical and field activities associated with soil, sediment, surface water, and biota samples to better characterize potential site risks and examine factors that influence metal bioavailability. Chemicals of potential concern included phthalates, PAHs, pesticides, PCBs, PCDD/Fs, metals, and cyanide. Performed laboratory and data validation coordination as well as review of sample receipt variances, laboratory quality control variances, analytical corrective actions, data verification issues (e.g., incomplete records), and data review corrective actions.

RI/FS Waiau Generating Station Honolulu, Hawai'i—Assisted with QAPP preparation for analytical and field activities for multiple sampling phases including onshore source investigation,

sediment transport evaluation, biota sampling, source control investigation, and pipe and tunnel investigation. Coordinated with analytical laboratories and data validation firm. Reviewed data validation reports.

Groundwater Monitoring and Delineation of Impacted Soil at Former Mill Site, Centralia, Washington—Coordinated laboratory analytical proposals and work orders, performed review of laboratory deliverables and tabular data, and assisted with field sampling planning.

Per- and Polyfluoroalkyl Substances, Northeastern U.S.—Serving as project chemist overseeing analyses and validation of per- and polyfluoroalkyl substances (PFAS) in groundwater, drinking water, surface water, soil, sediment, and porewater. Review isomer profiles of PFAS samples.

Kenmore Navigation Channel Sediment Characterization, Kenmore, Washington—Under a subcontract, prepared QAPP and health and safety plan (HASP) for a sediment characterization in 2019 to support maintenance dredging. Assisted with development of SAP and sample collection effort.

Elliott Bay Bioaccumulation Study, Seattle, Washington—Under a subcontract, prepared QAPP and HASP for the collection of surface sediment in 2019 to support a benchmark bioaccumulation study. Assisted with development of SAP and sample collection effort.

Ecological Baseline Pre-Design Investigation, Centredale Manor Restoration Project Superfund Site, Rhode Island—Prepared Uniform Federal Policy QAPP and performed quality assurance chemistry tasks in support of pre-remedial design investigation activities including ecological surveys and sampling stations, sampling equipment and procedures, sample designation, and sample handling. This Superfund site, based in North Providence, has multiple operating units. The site is associated with human health issues and ecological concerns from the presence of dioxins, furans, PCBs, pesticides, herbicides, and VOCs in all environmental media, but particularly in riverine and aquatic environments, associated biota, and floodplain soils.

Detailed Sediment Investigation, San Diego, California—Quality assurance chemistry in support of sediment investigation at two shipyards in San Diego Bay, focusing on the effects of metals, organo-metallic compounds, PAH, PCBs, polychlorinated triphenyls, and petroleum hydrocarbons on aquatic life, aquatic-dependent wildlife, and human health. Managed laboratory and data validation subcontracting.

Data Management and Validation

Deepwater Horizon Oil Spill, Natural Resource Damage Assessment—Worked in conjunction with the natural resource damage assessment team responding to the Deepwater Horizon accident and oil spill in the Gulf of Mexico on behalf of BP Exploration & Production Inc. Provided chemistry support and performed data validation and review of data validation reports associated with the environmental sample collection activities.

Industrial Site Data Validation, Vancouver, Washington—Performed data validation for a project involving the presence of chlorinated solvents at an active manufacturing facility in Vancouver, Washington. Project included groundwater monitoring and nearby residential air sample analyses, which are being used by the Washington State Department of Ecology for human health risk assessment.

Electrical Equipment Repair Facility Site Investigation Data Validation and Data Quality Assessment, Oregon—Performed data validation, data review, and data quality assessment for the site investigation of historical PCB releases at an electrical equipment inspection, service, and repair facility. The site was identified by the Oregon Department of Environmental Quality as a potential source of PCBs detected in the public stormwater system and in Willamette River sediments.

Groundwater Monitoring Program Data Validation, Beaverton, Oregon—Performed validation of groundwater chemistry results generated as part of a RCRA Corrective Action Program. Monitoring required for the project included VOCs and Appendix IX List compounds.

Fort Lewis Thermal Remediation Project Data Review and Validation, Fort Lewis, Washington—Performed chemical data review and validation on project data, including water and air samples for hydrocarbon and VOC analyses, using GC/photoionization detector and GC/MS, for a remediation project at Fort Lewis using electric resistance heating. The project was designed by the U.S. Army Corps of Engineers to be performed using near-real-time data from a mobile laboratory to make decisions about the remediation process using the Triad Approach.

Field Investigation Oversight and Report Preparation for a Coal-Fired Electrical Power Plant, Indiana—Performed data validation for a large environmental investigation of a coal-fired power plant. Data included groundwater, soils, and plant tissues.

Interim Remedial Actions/PCB Soil Removals, Cape Canaveral Air Force Station, Brevard County, Florida—Performed data validation and data assessment for a RCRA interim measures delineation and cleanup effort at Space Launch Complex 40 at Cape Canaveral Air Station, Florida. The project involved delineating TSCA levels in soil to determine PCB concentrations >50 ppm.

Voluntary Property Assessment (VPA) Activities, Former Crosstie Chipping Facility, Alabama—Performed data validation and data assessment for VPA investigation activities. Work included collection of numerous soil, sediment, surface water, groundwater, and macroinvertebrate samples to evaluate the extent of PAH impacts to the site and surrounding areas resulting from former crosstie chipping operations.

Former Truck Manufacturing Facility Remediation Data Validation and Data Quality Assessment, Washington—Performed data validation, data review, and data quality assessment for remediation of a former truck manufacturing facility located adjacent to the Duwamish River. The project work consisted of the collection of stormwater and tidal sediments.

Memphis Air National Guard, Memphis, Tennessee—Performed data quality review and data assessment on VOC data from the risk assessment and remediation of petroleum-impacted soil and groundwater.

White Swan Cleaners/Sun Cleaners Superfund Site, New Jersey—Performed data validation on CLP data, and data quality review and assessment on the data for ongoing collection activities related to a Settlement Agreement with EPA Region 2 to conduct an RI/FS of a regional site that has been contaminated by the dry cleaning solvent PCE. PCE had potentially impacted municipal water supply wells at a popular shoreline resort community.

Former Pharmaceuticals Facility Data Validation, Oregon—Performed data validation on the results related to the release of VOCs on the site. The primary contaminants of concern included trichloroethene, *cis*-1, 2-dichloroethene, and vinyl chloride, which were found at concentrations indicative of dense non-aqueous phase liquid.

Former Industrial Site Water Sampling Data Validation and Data Quality Assessment, New Jersey—Performed data validation, data review, and data quality assessment on the annual drinking water sampling at all homes surrounding a former industrial site, where the chemicals of concern in groundwater include VOCs—primarily 1,1,1-trichloroethane, 1,1-dichloroethylene, and 1,1-dichloroethane.

Groundwater and Surface Water Monitoring, Naval Facilities Engineering Command (NAVFAC), Fort Gordon, Georgia—Performed data validation, data review, and data quality assessment on quarterly groundwater sampling. Quarterly monitoring of groundwater and surface water was performed under a NAVFAC contract in compliance with NPDES for a wastewater treatment facility and land-application system at the Pointes West Army Recreation Area in Columbia County, Georgia.

Site Characterization at Industrial Operation, Seattle, Washington—Performed data validation, data review, and data quality assessment on the soil boring and groundwater sampling at the site. Site activities included site characterization (i.e., field assessment, focused site characterization report, project management) at an industrial operation approximately 2.1 acres in size located in Seattle, Washington. The site was impacted with metals, PCBs, PAHs, TPH, and VOCs.

West Virginia Department of Environmental Protection Brownfield Sites Data Validation and Data Quality Assessment, West Virginia—Performed data validation, data review, and data quality assessment using EPA Region 3 modifications to CLP national functional guidelines associated with Phase I surface soil sampling and follow-up Phase II subsurface soil sampling, groundwater investigations, and surface water and sediment sampling at various brownfield sites throughout West Virginia.

Massachusetts Military Reservation Closure Data Validation, Cape Cod, Massachusetts—Validated data for samples submitted for explosives compounds analysis and perchlorate, which are associated with verification that post-excavation bottom soils and expansion area soils are

below established action levels in order to obtain closure determination for the CS-19 and CS-18 Source Area sites at the Massachusetts Military Reservation in Cape Cod. Soil samples from the expansion areas were collected using the multi-increment sampling approach proposed by Cold Regions Research and Engineering Laboratory.

Susanville Sawmill and Cogeneration Facility, Susanville, California—Performed expedited data validation and associated report writing associated with air, water, soil, and product samples collected during the overall scope of work, which included site investigations and remediation at the proposed treatment cell area and fuel and maintenance area.

Rosiclare Mine Site, Rosiclare, Illinois—Validated data associated with soil, sediment, and groundwater sampling and wrote data validation report for the RI/FS effort to clean up historical fluorspar mine tailings.

Rental Car Maintenance Facility, San Jose, California—Performed expedited data validation and report writing associated with samples collected during the overall scope of work, which included removal and disposal of underground storage tanks, an aboveground storage tank, below-ground hydraulic lifts, and a car wash structure.

Former Ashland Lease Area, Shoreham Facility, Minneapolis, Minnesota—Performed data validation of quarterly groundwater samples analyzed for anions, conventional parameters, and VOCs and report writing for the monitoring program for the four remedial actions currently under way at the site: soil vapor extraction, light nonaqueous phase liquid monitoring and recovery, till bioremediation, and outwash pump and treat.

Smeltertown Superfund Site OU1, Salida, Colorado—Validated data from groundwater samples analyzed for metals and wrote report for the annual groundwater monitoring program.

Chemical Distribution Facility, Santa Ana, California—Validated data resulting from semiannual groundwater samples analyzed for PCE, TCE, chemical degradation products of PCE and TCE, and 1,4-dioxane and wrote data validation report as part of oversight of groundwater monitoring and soil remediation at the site.

Waste Rock Water Quality Assessment Open Pit Gold Mine Expansions, Nevada—Validated data associated with ongoing humidity cell test results of existing waste rock, alluvium, and drill cores of expansion material. Assisted with the quality assurance report associated with the 20-week results of the first round of humidity cell tests.

Former DDT Manufacturing Facility, Portland, Oregon—Validated data associated with stormwater monitoring at a former pesticide manufacturing facility under the jurisdiction of the Oregon Department of Environmental Quality. Also monitored laboratories' progress on sample analyses and reviewed and validated analytical results.

Blackwell Zinc Site, Blackwell, Oklahoma—Validated data associated with mitigation strategies of metals loading to the city's wastewater treatment plant resulting from infiltration of contaminated groundwater to the city's sanitary collection system.

Soil and Groundwater Investigation at Former Allied Engineering Facility, Alameda, California—Validated historical data and recent data associated with assessment and potential remediation of groundwater and sediment at the site.

Slag and Sewage Site, Past Costs and River Sediment Evaluation, Fox Point Park, Wilmington, Delaware—Performed Stage 2B and Stage 3 data validation associated with the sediment RI/FS in the Delaware River.

Hazardous Materials Assessment of Soils at Various Public Schools, Hawaii—Performed laboratory coordination and Stage 2B data validation associated with environmental hazard screening of select school sites for arsenic, lead, and organochlorine pesticides.

Former Wood Treating CERCLA Facility, Columbus, Mississippi—Performed data validation in support of a human health risk assessment, Operable Unit 1 focused feasibility study, and Operable Unit 1 removal action work plan, as well as implementation of the Operable Unit 1 voluntary removal action at a Superfund site.

Matthew E. Behum
Senior Scientist

Education and Credentials

M.S., Marine Science, University of South Carolina, Columbia, South Carolina, 2004

B.A., Environmental Biology (with Honors), Colgate University, Hamilton, New York, 2002

Certified Senior Ecologist, Ecological Society of America (2015)

Continuing Education and Training

SafeStart Certified Instructor (2019)

OSHA 10-Hour General Safety Training (2020)

Hazardous Waste Operations and Emergency Response 40-Hour Certification (2005; refresher 2020)

Hazardous Waste Operations Management and Supervisor 8-Hour Certification (2014)

Delaware Valley Safety Council Basic Orientation Plus Safety Certification (2015)

First Aid, CPR, and AED Certified (2020)

Risk-Based Corrective Action at Petroleum Release Sites (2005)

Professional Affiliations

Ecological Society of America

Society for Environmental Toxicology and Chemistry, Chesapeake-Potomac Regional Chapter

American Society of Safety Engineers

Achievements and Awards

Aqua Survey, Inc. Blue Peter Award for Environmental Remediation Leadership (2018)

Professional Profile

Mr. Matthew Behum is an ecologist with 15 years of experience in the field supporting multimedia ecological and human health risk assessments at Superfund sites and private industrial facilities. He has also performed environmental compliance audits with Maryland Vehicle Administration testing facilities. In addition to his risk assessment experience, Mr. Behum has sampled plants, benthic invertebrates, and aquatic vertebrates in both marine and freshwater systems and has identified juvenile invertebrates using molecular techniques. He has experience in ecological theory, database management, and general biostatistical analyses. He also has field management experience on a variety of projects, including coordination of a million-dollar, multiphase field effort involving biotic and abiotic sampling at a New Jersey Superfund site, as well as leading a data collection investigation of perfluoroalkyl substances (PFAS) in water systems on behalf of an East Coast chemical manufacturer. Mr. Behum is the office manager for Integral's Annapolis, Maryland, location, and is the company corporate health and safety manager.

Relevant Experience

Corporate Health and Safety Management

Prepared comprehensive revision of company health and safety program plan and coordinated its production. Regularly review all site health and safety plans prior to field operations. Manage health and safety credentials for all staff (e.g., Hazardous Waste Operations and Emergency Response, first aid, physicals, drug and alcohol testing, project-specific safety training). Utilize ISNet, Avetta, and BROWZ platforms for client-specific health and safety questionnaires, insurance updates, and project safety training coordination. Responsible for field safety updates, including job hazard analysis use, subcontractor prequalification questionnaires, and field auditing. Conduct company-wide presentations on safety topics and company safety updates. Also instituted the SafeStart safety program for all employees and currently manages that program for Integral.

Database Management and Statistical Analysis

Experience includes parametric and nonparametric statistical analyses. Project work has involved linear and multilinear

200 Harry S. Truman Parkway
Suite 330
Annapolis, MD 21401

410.573.1982, ext. 512
mbehum@integral-corp.com

regression investigations. Analytical platforms include Excel, Statistica, R, ProUCL, and GPOWER. Project experience includes working with relational databases using Access.

Environmental Auditing

Motor Vehicle Administration Sites, Maryland—Assisted with and led comprehensive voluntary audits of motor vehicle administration facilities to ensure compliance with multiple federal criteria including Clean Air Act, Clean Water Act, FIFRA, RCRA, and TSCA. Completed federal compliance checklists and drafted facility reports for client review. In addition, reviewed relevant state permitting policies, including whether relevant motor vehicle administration facilities were compliant for discharges from small municipal separate storm sewer systems (MS4s) within NPDES.

Environmental and Biological Sampling

Chemical Manufacturer, East Coast—Overseeing a multiphase data collection program to evaluate the presence of PFAS adjacent to a New Jersey chemical manufacturer. Managed subcontractors, budget, and the development of a work plan and field sampling plan. Initiated a data collection program, including seasonal public water supply sampling of various municipalities, private well sampling (including community outreach), temporary groundwater well sampling, onsite and offsite groundwater well sampling, surface water and sediment sampling of the Delaware and Schuylkill rivers, and onsite soil sampling.

Berry's Creek Superfund Site, Meadowlands, New Jersey—Completed multiple field surveys of Berry's Creek and surrounding tributaries of the Hackensack River, including water quality analyses of candidate reference sites to Berry's Creek and aquatic fauna surveys of the creek, in support of an RI/FS. Responsible for data syntheses of reference site surveys. Participated in marsh sediment sampling along floodplain transects and collected terrestrial and aquatic insects in surficial marsh sediment and *Phragmites* leaf litter for qualitative identification. Coordinated, managed, and participated in field sampling plans from 2010 to present for a variety of taxa in Berry's Creek and surrounding tributaries. Effort included oversight of budget, coordination with field staff (colleagues and teaming partners), along with refinement updates to standard operating procedures, field sampling plans, and work plan addenda. Recent field efforts include mercury air monitoring (including light intensity and temperature readings), fish residue biomonitoring collection, and marsh insect collection for residue analysis using various techniques, as well as benthic residue collection of annelids and fiddler crabs.

Curtis Bay Site, Curtis Bay, Maryland—Performed biota reconnaissance of nearshore sediment onsite at a former agricultural chemical production facility. Sediment samples were sieved and analyzed for invertebrate presence and species composition.

Portland Harbor Superfund Site, Portland, Oregon—Conducted crayfish sampling and sculpin longlining and electrofishing in Portland Harbor in support of multiclient remedial investigation.

Field Sampling, Central New York and Coastal South Carolina—Collected stream macroinvertebrates to assess community dynamics in response to changing riparian cover.

Collected fiddler crab juveniles and larvae in a salt marsh system to assess settlement patterns of two species.

Risk Assessment

Portland Harbor Superfund Site, Portland, Oregon—Analyzed sediment and fish data usability regarding N-qualified PCB and DDx data; performed syntheses of exploratory statistics addressing data usability concerns. Also performed multiple reviews of fish, plant, and amphibian baseline ecological risk assessments spanning multiple lines of evidence (e.g., tissue concentrations, surface water concentrations, and modeled effects). Performed exploratory biota-sediment accumulation factor calculations for whole-body smallmouth bass samples to compare with proposed findings of proposed Gobas model.

Yerington Mine Site, Yerington, Nevada—Developed mitigation plan for avian deterrence from site pumpback and evaporation ponds. Researched avian deterrence measures, consulted with experts, and helped to finalize a three-tiered approach involving amplified distress/predatory calls, pyrotechnics, and nonlethal projectiles. Managed wildlife observation database of the former Yerington mine site. Prepared quarterly reports. Monitoring was conducted to evaluate wildlife use at the site in support of ecological risk assessment.

Groundwater to Surface Water Interaction, Patrick Bayou, Texas—Managed groundwater, sediment, and ecotoxicological data to support weight-of-evidence evaluations of the impact of groundwater discharge to benthic communities. Also extensively researched Texas state ecological risk regulations.

Ecological and Human Risk Assessments, Formerly Used Defense Sites, Northeast U.S.—Participated in preparing screening level ecological and human health risk assessments of various media for multiple receptors at numerous formerly used defense sites. Conducted research of federal and secondary benchmarks for use in the screening-level risk assessments and proposed alternative screening values for ecological screening based on equilibrium partitioning theory.

Berry's Creek Superfund Site, New Jersey—As part of a proposal effort, evaluated food web dynamics of representative biota and plants exposed to mercury contamination in the Hackensack Meadowlands. Assisted in development of multi-tiered conceptual site model across various media in the creek. Also served as lead in a Phase 1 screening-level ecological risk assessment using published screening values for sediment, surface water, and wildlife tissue.

Curtis Bay Site, Curtis Bay, Maryland—Performed screening-level risk assessment of porewater exposures of volatile and semivolatile organic compounds to benthic invertebrates using ambient water quality criteria developed via secondary chronic values. Applied EPA methodology and narcosis theory based on bioavailability.

Greens Bayou, ISK Pond, Houston, Texas—Performed thorough research of appropriate literature for use in developing alternative benchmarks that were accepted by Trustees to support site

closure. Prepared a habitat equivalency analysis of sediment contamination as part of a natural resource damage assessment.

Upper Columbia River RI/FS, Washington—Prepared air data statistical summaries of beach dust monitoring in support of a remedial investigation. Used Statistica and ProUCL platform applications.

Regional Risk Assessment of a River Estuary, Delaware—Composed summaries of physical, chemical, and biological stressors affecting tidal stretch of Delaware estuary, which were then used for regional risk assessment. Managed junior staff investigating stressor identification. Updated reference database for project.

Exxon Valdez Oil Spill, Prince William Sound, Alaska—Assisted in researching and compiling literature related to natural resource injury associated with the 1989 *Exxon Valdez* oil spill.

Risk Assessment of West Nile Virus Incidence and Control, Suffolk County, New York—Conducted statistical analyses of air concentrations for various pesticides and synergist chemicals portraying statistical results for use in the risk assessment. Assisted in the evaluation of ecological risks associated with mosquito-control activities.

Research

Chesapeake Bay Environmental Issues—Conduct regular research of environmental and policy issues facing Chesapeake Bay and involving all states that are part of its watershed. Provide updated information to colleagues on an ongoing basis.

Water Quality Criteria Research—Engage in extensive research of federal and secondary benchmark development from a variety of sources. Focus on understanding how benchmarks are developed using both conventional and equilibrium partitioning approaches. Have obtained extensive knowledge of ambient water quality criteria data requirements.

Macroinvertebrate Survey along a Stream Continuum, Central New York—Conducted field sampling of macroinvertebrates and identified them to functional feeding group and genera to analyze shifting feeding groups in response to dynamic stream canopies. Collected fine and coarse particulate organic matter samples along with chlorophyll *a* concentrations at all sampling locations. Results presented for honors degree in environmental biology.

Postlarval Settlement Patterns of Fiddler Crabs across Salt Marsh Habitats, Winyah Bay Estuary Marshes, South Carolina—Conducted field sampling of juvenile and larval fiddler crabs, identifying them to species using restriction fragment length polymorphism molecular techniques. Oversaw undergraduate interns assisting in laboratory and fieldwork. Also analyzed surface sediment temperature and moisture. Results documented and presented at 2004 Benthic Ecology Meeting and published in *Marine Ecology Progress Series*.

Wind Power Business Development—Led initiatives to incorporate population-level risk assessment techniques, including GIS encounter modeling with bird and bat strikes, probabilistic risk assessment, and preferred bird and bat habitat analyses with wind power development.

Publications

Behum, M.E., R.J. Brodie, and J.L. Staton. 2005. Distribution of juvenile *Uca pugnax* and *U. pugilator* across habitats in a South Carolina estuary, assessed by molecular techniques. *Mar. Ecol. Prog. Ser.* 288:211–220.

Brodie, R.J., M.E. Behum, E. Monroe, N. Glenn, and J.L. Staton. 2005. Recruitment to adult habitats following marine planktonic development in the fiddler crabs, *Uca pugilator*, *U. pugnax*, and *U. minax*. *Mar. Biol.* 147:105–111.

Presentations / Posters

Behum, M. 2018. Challenges presented by new, temporary, and young employees. Platform presentation, Retia Safety Forum Invitational, Nashville, TN. September 18–19.

Behum, M. 2017. Safety metrics, best practices, and lessons learned: Safety culture in a small firm. Platform presentation, Retia Safety Forum Invitational, Nashville, TN. September 20–21.

Behum, M. 2016. Safety metrics, best practices, and lessons learned: Office safety. Platform presentation, Retia Safety Forum Invitational, Nashville, TN. September 14–15.

Behum, M., J. Durda, D. Himmelheber, and P. Brussock. 2015. Camera surveys to document human use in an isolated urban estuary: Update and analysis. Poster presentation, Eighth International Conference on Remediation and Management of Contaminated Sediments, New Orleans, LA. January 12–15.

Behum, M., J. Lape, J. Durda, P. de Haven, and J. Wollenberg. 2015. Air monitoring in a mercury-contaminated estuary: Support for risk assessment and risk management. Poster presentation, Eighth International Conference on Remediation and Management of Contaminated Sediments, New Orleans, LA. January 12–15.

Durda, J., M. Behum, P. de Haven, and J. Wollenberg. 2015. Physical and ecological conditions in marshes: Exposure pathways, assessment, and implications for risk management. Platform presentation, Eighth International Conference on Remediation and Management of Contaminated Sediments, New Orleans, LA. January 12–15.

Behum, M., J.L. Durda, and J. Samuelian. 2013. Marsh invertebrate community surveys to support ecological risk assessment in a New Jersey estuary. Poster presentation, Seventh International Conference on Remediation of Contaminated Sediments, Dallas, TX. February 4–7.

Pastorok, R.A., D.V. Preziosi, and M.E. Behum. 2012. The role of population modeling in risk assessment at wind energy facilities. Poster presentation, National Wind Coordinating Collaborative Wind Wildlife Research Meeting, Broomfield, CO. November 27–30.

Behum, M.E. 2010. Unique approach to assessing wildlife population risk from wind turbine development. Platform presentation, Chesapeake-Potomac Regional Chapter Meeting of the Society of Ecotoxicology and Chemistry, Towson, MD. April 19.

Behum, M.E. 2009. Fostering career development and mentoring. Platform presentation, Integral Consulting Inc. company retreat, Stevenson, WA. May 16.

Behum, M.E. 2004. Postlarval settlement patterns of fiddler crabs across salt marsh habitats. Platform presentation, 33rd Annual Marine Benthic Ecology Meeting, Mobile, AL. March 25–28.

Craig Hutchings
Consultant

Education and Credentials

B.S., Environmental Studies, The Evergreen State College, Olympia, Washington, 1991

Professional Profile

Craig Hutchings is a chemist with more than 25 years of experience in environmental investigations, environmental analytical chemistry, and QA/QC data review and validation. He is responsible for the preparation of sampling and analysis plans (SAPs) and quality assurance project plans (QAPPs), data interpretation, and development of quality assurance programs for sites within various state and federal regulatory programs. Mr. Hutchings has coordinated communication between laboratories and data users on several projects involving performance-based methods for non-standard analytes and methods to ensure that methodologies, data quality, and deliverables meet project needs. Mr. Hutchings has validated both the newly developed methodologies used for these projects and the data generated. He has authored numerous SAPs and QAPPs that comply with federal or state regulatory programs and take into account unique data quality objective requirements of specific projects. Mr. Hutchings has performed and supervised gas chromatography, high performance liquid chromatography, and gas chromatography/mass spectrometry analyses for contaminants in soil, sediment, water, and tissues using EPA and various state methods. He is experienced in the evaluation and review of analytical data including outputs from inductively coupled plasma instruments and chromatograms for Aroclors, pesticides, and other compounds and has reviewed chemistry data from numerous projects involving a wide variety of analyses in air, water, soils, sediments, and tissues.

Relevant Experience

Per- and Polyfluoroalkyl Substances, Northeastern U.S.—Serving as project chemist overseeing analyses and validation of per- and polyfluoroalkyl substances (PFAS) in groundwater, drinking water, surface water, soil, sediment, and pore water. Review isomer profiles of PFAS samples and assist in investigations of potential third-party source contributions.

Yosemite Slough, Technical Pre-Design Studies, San Francisco, California—Developed QAPP for technical pre-design studies related to design of non-time critical removal action. The QAPP addressed analyses of sediment, pore water, and surface water for chemical and physical parameters to inform cap design and studies

1205 West Bay Drive NW
Olympia, WA 98502

360.705.3534, ext. 417
chutchings@integral-corp.com

of sediment resuspension, hydrodynamics, and the potential use of monitored and enhanced natural attenuation as an alternative to capping or dredging.

Third-Party Data Integrity Evaluation, Confidential Location—Worked with project team to perform a detailed third-party review of 4 years of chemistry data to investigate allegations of improper practices and data falsification at a small-scale wastewater treatment plant laboratory. Examined hard copy and electronic data and ancillary documents to evaluate the validity of analytical results and conformity with analytical methods, laboratory standard operating procedures, and best laboratory practices. Assisted in preparation of a technical report summarizing the evaluation techniques and conclusions to support reporting by legal counsel to the state regulatory agency.

Biomonitoring Study, U.S.—Served as project manager for a biomonitoring study conducted in support of litigation. Coordinated study design and implementation, laboratory oversight, and data management. Evaluated laboratory methodologies, provided laboratory oversight and coordination, evaluated data usability, and prepared a report summarizing the data and results.

Third-Party Analytical Data Quality Review, Confidential Location—Worked with project team to provided extensive third party review of 8 years of analytical chemistry data records from three laboratory instruments to identify and evaluate the impacts of improper laboratory practices. Examined raw instrument files, laboratory data packages, hard copy documentation, and the laboratory's information management system database to assess conformity with analytical methods, laboratory standard operating procedures, and best laboratory practices.

Deepwater Horizon, Gulf of Mexico—Worked in conjunction with the consulting team in responding to the *Deepwater Horizon* accident and oil spill in the Gulf of Mexico on behalf of BP Exploration & Production Inc. Provided quality assurance and offshore sample coordination roles to support sample collection, data management, and reporting activities for multiple technical work groups. Participated in the chemistry technical working group in review of the quality assurance plan, laboratory coordination, and other quality assurance review activities and data completeness tasks.

Regional-Scale Risk Assessment, Former Mill and Mine Sites, Illinois—Led the review and data validation of analyses of soil, sediment, and surface water for lead, cadmium, chromium, zinc, mercury, and cyanide.

San Jacinto River Waste Pits RI/FS, Houston, Texas—Project chemist for a remedial investigation of dioxin contaminated sediments. Assisted in the development of quality assurance project plans for sediment, soil, and tissue investigations. Performed laboratory coordination for all aspects of the investigation—soil, sediment, groundwater, and tissue.

Upper Columbia River RI/FS, Washington and British Columbia—Project chemist for a remedial investigation of the upper Columbia River. Carried primary responsibility for the development of quality assurance project plan sections related to laboratory activities, the preparation of a

comprehensive laboratory request for proposal, and the selection of laboratories for approximately $3 million of analyses. Performed laboratory coordination for beach sediment sampling events.

Portland Harbor RI/FS, Portland, Oregon—Provided assistance to lead project chemists and task managers for this ongoing remedial investigation of a 9-mile stretch of industrialized, urban river. Reviewed analytical data and chromatograms to resolve technical issues, including reviews of chromatograms to confirm Aroclor identifications and the effects of Aroclors on pesticide identifications. Reviewed historical chemistry data for stormwater and managed data to be added to the project database. Completed data validation for a wide variety of analyses, including dioxins and furans, PCB congeners, and EPA methods in sediment, tissue, surface water, and transition zone water samples.

Presentations / Posters

Luz, A., C. Hutchings, J. Anderson, P. Goodrum, and J. Field. 2019. A novel approach for assessing hazard associated with firefighting foams. Poster presentation at SETAC North America 40th Annual Meeting, Toronto, Ontario, Canada. November 3–7.

Goodrum, P., A. Luz, J. Anderson, G. Ansell, and C. Hutchings. 2019. Approaches for perfluoroalkyl acid grouping and assessment of mixture toxicity. SETAC North America Focused Topic Meeting: Environmental Risk Assessment of PFAS, Durham, NC. August 12–15.

Hutchings, C., and S. Helgen. 2019. Identifying linear and branched isomers from standard PFAS analysis for source delineation. Platform presentation at Tenth International Conference on the Remediation and Management of Contaminated Sediments, New Orleans, LA. February 11–14.

Helgen, S., M. Marietta, C. Hutchings, and E. Palko. 2018. Site-specific desorption testing of perfluorononanoic acid (PFNA) to assess potential soil leaching to groundwater. Platform presentation at Eleventh International Conference on Remediation of Chlorinated and Recalcitrant Compounds, Palm Springs, CA. April 8–12.

Jones, L., and C. Hutchings. 2013. Superfund data validation. Lorman Education Services Audio Conference.

Hutchings, C., and A. Bailey. 2006. Variables in lipids analyses and effect on data quality. 22nd National Environmental Monitoring Conference, Arlington, VA.

Bailey, A.K., P. Kane, and C. Hutchings. 2006. Reference materials as indicators of analytical data quality for human and ecological risk assessments. Tenth International Symposium on Biological and Environmental Reference Materials, Charleston, SC.

Manon Tanner-Dave
Project Scientist

Education and Credentials

M.S., Environmental Science, Oregon Health & Science University, Portland, Oregon, 2002

B.S., Chemistry, Pacific University, Forest Grove, Oregon, 2001

A.A., General, Modesto Junior College, Modesto, California, 1998

Continuing Education and Training

Confined Space Entry Awareness Course (2007)

Red Cross CPR (2007) and First Aid Training (2004)

Hazardous Waste Operations and Emergency Response 40-Hour Certification (2004; refreshers current)

Oregon Department of Transportation Training (2009)

Professional Profile

Ms. Manon Tanner-Dave is a chemist with 15 years of experience providing support in project and data quality assurance. She has extensive experience writing field sampling plans and quality assurance project plans (QAPPs) related to a variety environmental media for both state and federal regulatory agencies, including Alaska Department of Environmental Conservation, Washington State Department of Ecology, EPA Regions 6 and 10, and the U.S. Army Corps of Engineers. She specializes in environmental chemistry and is experienced in data validation for organic and inorganic data using EPA's functional guidelines for data validation. She has coordinated analytical laboratory activities and works closely with clients, project teams, and laboratories to resolve any data quality issues, such as background contamination or analytical interference. In addition, Ms. Tanner-Dave is knowledgeable of many analytical methods for environmental matrices, including EPA SW-846, Standard Methods for the Examination of Water and Wastewater, and ASTM International standards.

Relevant Experience

Portland Harbor Superfund Site Remedial Investigation/Feasibility Study, Portland, Oregon—Assisted in the development of QAPPs and several QAPP addenda for an extensive list of organic and inorganic analytes of interest in soil, sediment, tissue, surface water, and groundwater. Prepared letters of authorization to participating analytical laboratories and data validation firms and assisted with budget projections. Coordinated and oversaw all analytical laboratory services and assisted in sample collection and shipment of samples to the analytical laboratories. Worked closely with the analytical laboratories on analytical method modifications needed for some problematic environmental matrices. Evaluated data and assisted in preparing field sampling reports and data quality assurance reports.

Blood Lead Biomonitoring Study, Rico, Colorado—Assisted in developing a biomonitoring study for a former lead mining town in Colorado. Prepared a QAPP for the study, coordinated field activities with the analytical laboratories, validated analytical results, and prepared a quality assurance data report. Sampled media included blood, house dust, drinking water, and paint. Results of

319 SW Washington Street
Suite 1150
Portland, OR 97204

503.943.3616
mtanner@integral-corp.com

this study were used to evaluate seasonal fluctuations of blood lead levels in town residents and to assess the effectiveness of soil remediation efforts.

Former Chemical Manufacturing Facility, Portland, Oregon—Drafted QAPPs in support of interim and remedial measures for stormwater as well as post-construction stormwater monitoring for site-specific organic and inorganic analytes of concern. Coordinated laboratory analyses of samples. Reviewed and validated analytical results from stormwater interim and remedial measures, and post-construction stormwater monitoring of site-specific organic and inorganic analytes of concern. Prepared data quality assurance reports summarizing data results.

Post-construction Groundwater Monitoring Program, Smeltertown Superfund Site, Operable Unit No. 1, Salida, Colorado—Provided data validation services for a groundwater monitoring project. Analytes of interest included select metals and semivolatile organic compounds. Provided a data validation summary report.

Subslab Gas Sampling, Milwaukie International Way Site, Milwaukie, Oregon—Participated in semiannual subslab gas sampling Soil gas probes were installed and sampled following EPA guidance, *Standard Operating Procedure (SOP) for Installation of Sub-Slab Vapor Probes and Sampling Using EPA Method TO-15 to Support Vapor Intrusion Investigations*. Reviewed and validated analytical results from subslab air samples that were collected and analyzed according to EPA Method TO-15. Provided a data quality summary report for each sampling event.

Human Health Risk Assessment at Smelter Facility, La Oroya, Peru—Assisted in developing a human health risk assessment for an active smelter. Prepared a study QAPP, coordinated field activities with the analytical laboratories (international and domestic), validated analytical results, and prepared a quality assurance data report. Also participated in one of two field sampling efforts in the community surrounding the smelter to characterize exposure media, including drinking water, surface soil, outdoor dust, and dust in homes. These data were combined with biomonitoring and dietary intake data for the population and air dispersion and deposition modeling results to complete the human health risk assessment. For this project all communications occurred in Spanish.

Semiannual Groundwater Sampling at a Former Wood Waste Disposal Landfill, Oakridge, Oregon—Collected groundwater and surface water samples at a former wood waste landfill site. Analyzed field and analytical data to modify an existing environmental monitoring plan for the site that included a reduced list of analytes.

Water Quality Monitoring for Mining Site, Ketchikan, Alaska—Prepared QAPPs in support of routine monitoring of surface water and groundwater quality, and quarterly monitoring of the wastewater treatment facility effluent for the site barge, which houses onsite employees. Reviewed and validated analytical results from quarterly groundwater, surface water, and wastewater treatment facility effluent from the site barge. Assisted with quarterly and annual data evaluation, reporting, and development of a database for analytical results.

Volcanogenic Massive Sulfide Project, Alaska—Prepared a QAPP using Alaska Department of Environmental Conservation guidance in support of assembling a sufficient data set to define baseline conditions for target analytes in surface water. Analytes of interest included conventional parameters, cations/anions, and total/dissolved metals. Baseline water quality data were used to characterize water quality typical of the project area prior to any potential underground exploration or mine development. Monitored laboratories' progress on sample analyses. Reviewed and validated analytical results from baseline water quality testing. Provided data quality reports summarizing analytical results and assisted with development of a database for analytical results.

Glenbrook Nickel Site, Coos Bay, Oregon—Provided data validation services for sediment samples submitted for total organic carbon, grain size, and nickel analyses for site investigation purposes. Responsible for providing a data quality summary report of all analytical results.

Groundwater and Wastewater Sample Data Validation, Blackwell, Oklahoma—Reviewed and validated total and dissolved cadmium, lead, zinc, calcium, and magnesium results from groundwater samples. Reviewed and validated cadmium, lead, and zinc results from wastewater treatment plant influent samples. Reviewed and validated cadmium, lead, zinc, naphthalene, and select volatile organic compound results from groundwater treatment facility samples. Assisted with quarterly and annual reporting of analytical results.

Formerly Used Defense Sites—Performed data validation for munitions-related constituents. Provided data validation reports for each area of concern. Delegated role as lead data validator and performed senior review of data validation results and data validation reports.

Deepwater Horizon, Gulf of Mexico—Worked in conjunction with the Cardno ENTRIX team in responding to the Deepwater Horizon accident and oil spill in the Gulf of Mexico on behalf of BP Exploration & Production Inc. Provided support to the chemistry technical working group in quality assurance review activities and data completeness tasks. Primarily validated data and provided data validation review support according to project-specific quality assurance plan specifications.

ATTACHMENT 3

CHAIN OF CUSTODY FORM

>>> Select a Laboratory <<<

#N/A
#N/A
#N/A
#N/A

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:

APPENDIX D

STANDARD OPERATING PROCEDURES

CHARACTERIZATION WORK PLAN:
STUDY AREA OPERABLE UNIT 4 FLOOD CONTROL AREAS

NYSDEC Project ID 851046, Corning, New York

Standard Operating Procedures

Prepared for
Corning Incorporated
Corning, NY

Prepared by

1001 6th Avenue
11th Floor
New York, NY 10018

November 12, 2020

Affiliated with Integral Consulting Inc.

All Purpose Standard Operating Procedures

- SOP AP-01 Sample Packaging and Shipping
- SOP AP-02 Field Documentation
- SOP AP-03 Sample Custody
- SOP AP-04 Sample Labeling
- SOP AP-05 Characterization Derived Waste Handling

Sediment Standard Operating Procedures

- SOP SD-01 Decontamination of Sediment Sampling Equipment
- SOP SD-02 Preparation of Field Quality Control Samples for Sediments
- SOP SD-08 Sediment Core Collection Using a Vibracorer
- SOP SD-11 Field Analyses for Sediments
- SOP SD-14 Sediment Coring Procedures Using a Wilner Corer

Soil Standard Operating Procedures

- SOP SL-01 Decontamination of Soil Sampling Equipment
- SOP SL-02 Preparation of Field Quality Control Samples for Soils
- SOP SL-04 Field Classification of Soil
- SOP SL-05 Surface Soil Sampling
- SOP SL-06 Logging of Soil Boreholes
- SOP SL-07 Subsurface Soil Sampling

Surface Water Standard Operating Procedures

- SOP SW-01 Decontamination of Surface Water Sampling Equipment
- SOP SW-20 Stormwater Grab Sampling

STANDARD OPERATING PROCEDURE (SOP) AP-01

SAMPLE PACKAGING AND SHIPPING

SCOPE AND APPLICATION

This SOP describes specific requirements for sample packaging and shipping to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein. This SOP also presents the method to be used when packing samples that will either be hand delivered or shipped by commercial carrier to the laboratory.

EQUIPMENT AND SUPPLIES REQUIRED

Make sure that you have the equipment and supplies necessary to properly pack and ship environmental samples, including the following:

- Project-specific sampling and analysis plan (SAP)
- Project-specific field logbook
- Sealable airtight bags in assorted sizes (e.g., Ziploc®)
- Wet ice in doubled, sealed bags; frozen Blue Ice®; or dry ice
- Cooler(s)
- Bubble wrap
- Fiber-reinforced packing tape, clear plastic packing tape, and duct tape
- Scissors or knife
- Chain-of-custody (COC) forms
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick)
- Paper towels
- "Fragile," "This End Up," or "Handle With Care" labels
- Mailing labels
- Air bills for overnight shipment

PROCEDURE

Customize the logistics for sample packaging and shipping to each study. If necessary, transfer samples from the field to a local storage facility where they can be frozen or refrigerated. Depending on the logistics of the operation, field personnel may transport samples to the laboratory or use a commercial courier or shipping service. In the latter case, Integral field personnel must be aware of any potentially limiting factors to timely shipping, such as availability of overnight service and weekend deliveries to specific areas, and shipping regulations regarding "restricted articles" (e.g., dry ice, formalin) prior to shipping the samples.

SAMPLE PREPARATION

Take the following steps to ensure the proper transfer of samples from the field to the laboratories:

At the sample collection location:

1. Document all samples using the proper logbooks or field forms (see SOP AP-02), required sample container identification (i.e., sample labels with tag numbers), and COC form (example provided in SOP AP-03). Fill out the COC form as described in SOP AP-03, and use the sample labeling techniques provided in SOP AP-04.
2. Make all applicable laboratory quality control sample designations on the COC forms. Clearly identify samples that will be archived for future possible analysis. Label these samples as follows: "Do Not Analyze: Hold and archive for possible future analysis." Some laboratories interpret "archive" to mean that they should continue holding the residual sample after analysis.
3. Notify the laboratory contact and the Integral project quality assurance/quality control (QA/QC) coordinator that samples will be shipped and the estimated arrival time. Send copies of all COC forms to Integral's project QA/QC coordinator or project manager, as appropriate.
4. Keep the samples in the possession of the sampling personnel at all times. Lock and secure any temporary on-location sample storage areas to maintain sample integrity and COC requirements.
5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
6. Complete the COC form as described in SOP AP-03, and retain the back (pink) copy for project records prior to sealing the cooler. Check sample containers against the COC form to ensure all the samples that were collected are in the cooler.

7. Store each sample container in a sealed plastic bag that allows the sample label (example provided in SOP AP-03) to be read. Before sealing the bags, ensure that volatile organic analyte (VOA) vials are encased in a foam sleeve or in bubble wrap.
8. If the samples require storage at a specific temperature, place enough ice in the sample cooler to maintain the temperature (e.g., 4°C) throughout the sampling day.

At the sample processing area (immediately after sample collection) take the following steps:

1. If the samples require a specific storage temperature, then cool the samples and maintain the temperature prior to shipping. For example, place enough ice in each sample cooler to maintain the temperature at 4°C until processing begins at the testing laboratory.
2. Be aware of holding time requirements for project-specific analytes and arrange the sample shipping schedule accordingly.
3. Place samples in secure storage (i.e., locked room or vehicle) or keep them in the possession of Integral sampling personnel before shipment. Lock and secure any sample storage areas to maintain sample integrity and COC requirements.
4. Store samples in the dark (e.g., keep coolers shut).

At the sample processing area (just prior to shipping), do the following:

1. Check sample containers against the COC form to account for all samples intended for shipment.
2. Choose cooler(s) of appropriate size and make sure they are clean of gross contamination inside and out. If the cooler has a drain, close the drain and secure it with duct tape.
3. Line the cooler with bubble wrap and place a large plastic bag (preferably with a thickness of 3 mil), open, inside the cooler.
4. Individually wrap each glass container (which was sealed in a plastic bag at the collection location) in bubble wrap and secure with tape or a rubber band. Place the wrapped samples in the large plastic bag in the cooler, leaving room for ice to keep the samples cold (i.e., 4°C).
5. If temperature blanks have been provided by the testing laboratory, place one temperature blank in each sample cooler.
6. If the samples require a specific storage temperature, add enough wet ice or Blue Ice® to maintain that temperature during overnight shipping (i.e., 4°C). Always overestimate the amount of ice that will be required. Keep ice in a sealed plastic bag, which is placed in a second sealed plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it may insulate the samples from the ice. After adding all samples and ice to the cooler, use bubble wrap (or other

available clean packing material) to fill any empty space and prevent the samples from shifting during transport.

7. If possible, consolidate all VOA samples in a single cooler and ship them with (a) trip blank(s) if the project-specific QA project plan calls for them.
8. Sign, date, and include any tracking numbers provided by the shipper on the COC form. Remove the back (pink) copy of the original COC form and retain this copy for the project records.
9. Seal the rest of the signed COC form in a bag and tape the bag to the inside of the cooler lid. Each cooler should contain an individual COC form for the samples contained inside it. If time is short and it becomes necessary to combine all the samples onto a single set of COC forms and ship multiple coolers together, then indicate on the outside of the appropriate cooler, "Chain-of-Custody Inside."
10. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it with fiber-reinforced packing tape. Tape the cooler around the opening, joining the lid to the bottom, and around the circumference of the cooler at both hinges.
11. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid (provided with example field forms). Place one seal on the front right portion of the cooler and one on the back left. Be sure the seals are properly affixed to the cooler to prevent removal during shipment. Additional tape across the seal may be necessary if the outside of the cooler is wet.

SAMPLE SHIPPING

Hand Delivery to the Testing Laboratory

1. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. When hand-delivering environmental samples, make sure the testing laboratory receives them on the same day that they were packed in the coolers.
3. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after it has been faxed. Never leave the original COC form in the custody of non-Integral staff.

Shipped by Commercial Carrier to the Laboratory

1. Apply a mailing label to the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care." If the shipment contains multiple coolers, indicate on the mailing label the number of coolers that the testing laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the cooler and to protect it from the weather. This is a secondary label in case the air bill is lost during shipment.
2. Fill out the air bill and fasten it to the handle tags provided by the shipper (or the top of the cooler if handle tags are not available).
3. If samples must be frozen (–20°C) during shipping, make sure that dry ice has been placed in the sample cooler. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require.
4. Make sure that benthic infauna samples have been preserved with formalin in the field prior to shipping. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require for these samples.
5. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be shipped and the estimated arrival date and time. If environmental samples must be shipped at 4°C or –20°C, choose overnight shipping for delivery next morning. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after faxing. Never leave the original COC form in the custody of non-Integral staff.

STANDARD OPERATING PROCEDURE (SOP) AP-02

FIELD DOCUMENTATION

SCOPE AND APPLICATION

This SOP describes the Integral procedure for accurate record-keeping in the field for the purposes of ensuring that samples can be traced from collection to final disposition.

Document all information relevant to field operations properly to ensure that activities are accounted for in written records to the extent that someone not present could reconstruct the activity without relying on the memory of the field crew. Several types of field documents are used for this purpose and should be consistently used by field personnel. Field documentation should include only a factual description of activities and observations. Field personnel should not include superfluous comments or speculation regarding the field activities or observations.

FIELD LOGBOOKS

During field sampling events, field logbooks must be used to record all daily activities. The purpose of the field logbook is to document events and record data measured in the field to the extent that someone not present could reconstruct the activity without relying on the memory of the field crew. The project manager (or designee) should issue a field logbook to the appropriate personnel for the direction of activities (e.g., reconnaissance survey team leader, sampling team leader). It is this designee's responsibility to maintain the logbook while it is in his or her possession and return it to the project manager or turn it over to another field team.

Make entries in the field logbook as follows:

1. Document all daily field activities in indelible ink in the logbook and make no erasures. Make corrections with a single line-out deletion, followed by the author's initials and the date. The author must initial and date each page of the field logbook. The author must sign and date the last page at the end of each day, and draw a line through any blank space remaining on the page below the last entry.

2. Write the project name, dates of the field work, study area name and location (city and state), and Integral job number on the cover of the field logbook. If more than one logbook is used during a single sampling event, then annotate the upper right-hand corner of the logbook (e.g., Volume 1 of 2, 2 of 2) to indicate the number of logbooks used during the field event. Secure all field logbooks when not in use in the field. The following is a list of the types of information that is appropriate for entry in the field notebook:
   - Project start date and end date
   - Date and time of entry (24-hour clock)
   - Time and duration of daily sampling activities
   - Weather conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change (e.g., wind speed and direction, rain, thunder, wave action, current, tide, vessel traffic, air and water temperature, thickness of ice if present)
   - Name and affiliation of person making entries and other field personnel and their duties, including what times they are present
   - The location and description of the work area, including sketches, map references, and photograph log, if appropriate
   - Level of personal protection being used
   - Visitors (names and affiliations), if any, including what times they are present
   - The name, agency, and telephone number of any field contacts
   - Notation of the coordinate system used to determine the station location
   - The sample identifier and analysis code for each sample to be submitted for laboratory analysis, if not included on separate field data sheets
   - All field measurements made (or reference to specific field data sheets used for this purpose), including the time of collection and the date of calibration, if appropriate
   - The sampling location name, date, gear, water depth (if applicable), and sampling location coordinates, if not included on separate field data sheets
   - For aquatic sampling, the type of vessel used (e.g., size, power, type of engine)
   - Specific information on each type of sampling activity
   - The sample type (e.g., groundwater, soil, surface sediment), sample number, sample tag number, and any preservatives used, if not included on separate field data sheets
   - Sample storage methods

- Cross-references of numbers for duplicate samples
- A description of the sample (source and appearance, such as soil or sediment type, color, texture, consistency, presence of biota or debris, presence of oily sheen, changes in sample characteristics with depth, presence/location/thickness of the redox potential discontinuity [RPD] layer, and odor) and penetration depth, if not included on separate field data sheets
- Estimate of length and appearance of recovered cores, if not included on separate field data sheets
- Photographs (uniquely identified) taken at the sampling location, if any
- Details of the work performed
- Variations, if any, from the project-specific sampling and analysis plan (SAP) or standard operating protocols and reasons for deviation
- Details pertaining to unusual events that might have occurred during sample collection (e.g., possible sources of sample contamination, equipment failure, unusual appearance of sample integrity, control of vertical descent of the sampling equipment)
- References to other logbooks or field forms used to record information (e.g., field data sheets, health and safety log)
- Any field results not appearing on the field data sheets (if used), including station identification and location, date, and time of measurement
- Sample shipment information (e.g., shipping manifests, chain-of-custody (COC) form numbers, carrier, air bill numbers, time addresses)
- A record of quantity of characterization-derived wastes (if any) and storage and handling procedures.

3. During the field day, as listed above, record in the logbook a summary of all activities. Provide a date and time for each entry. The information need not duplicate anything recorded in other field logbooks or field forms (e.g., health and safety officer's logbook, calibration logbook, field data sheets), but should summarize the contents of the other logbooks and refer to the pages in these logbooks for detailed information.
4. If measurements are made at any location, record the measurements and equipment used, or refer to the logbook and page number(s) or field forms on which they are recorded. All maintenance and calibration records for equipment should be traceable through field records to the person using the instrument and to the specific piece of instrumentation itself.

5. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided below.

FIELD DATA FORMS

Occasionally, additional field data forms are generated during a field sampling event (e.g., groundwater monitoring form, sediment core profile form, water quality measurement form) to record the relevant sample information collected. For instructions regarding the proper identification of field data forms, sampling personnel should consult the project-specific SAP.

Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field data forms to be copied. A discussion of copy distribution is provided below.

PHOTOGRAPHS

In certain cases, photographs (print or digital) of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Ensure that photographs include a measured scale in the image, when practical. If you take photographs of sample characteristics and routine sampling activities, avoid using telephoto or wide-angle shots, because they cannot be used in enforcement proceedings. Record the following items in the field logbook for each photograph taken:

1. The photographer's name or initials, the date, the time of the photograph, and the general direction faced (orientation)
2. A brief description of the subject and the field work shown in the picture
3. For print photographs, the sequential number of the photograph and the roll number on which it is contained
4. For digital photographs, the sequential number of the photograph, the file name, the file location, and back-up disk number (if applicable).

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all photographic materials to be developed (prints) or copied (disks). Place the prints or disks and associated negatives in the project files (at the Integral project manager's location). Make photocopies of photo logs and any supporting documentation from the field logbooks, and place them in the project files with the prints or disks.

EQUIPMENT CALIBRATION RECORDS

Record in the field logbook all equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration. Calibrate all equipment used daily, at a minimum, in accordance with the manufacturers' recommendations.

DISTRIBUTION OF COPIES

When the field team has returned from the sampling event, the field team leader is responsible for making sure that the field documentation is 1) scanned and placed into the project file on the portal (in a subfolder named Field under Working_Files), and 2) a copy of all field logbooks and additional field data forms is made and placed into the project file. Both the scanned copy and the hard copy will be available for general staff use.

The original field logbooks and forms will be placed in a locked file cabinet for safekeeping. One file cabinet at each Integral office will contain the original field documentation for multiple projects. The original field documentation will be filed at the Integral office where the project manager is located.

SET-UP OF LOCKING FILE CABINET

Place each project in its own file folder in a locking file cabinet. On the folder label, include the project name and contract number. Each project folder will include up to six kinds of files:

- Field logbook(s)
- Additional field data forms
- Photographs
- COC forms
- Acknowledgment of Sample Receipt forms
- Archive Record form (to be completed only if samples are archived at an Integral field storage facility or Integral laboratory).

STANDARD OPERATING PROCEDURE (SOP) AP-03

SAMPLE CUSTODY

SCOPE AND APPLICATION

This SOP describes Integral procedures for custody management of environmental samples.

A stringent, established program of sample chain-of-custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used with SOP AP-01, which covers sample packaging and shipping; SOP AP-02, which covers the use of field logbooks and other types of field documentation; and SOP AP-04, which covers sample labeling. Chain-of-custody (COC) forms ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession
2. The sample is in the person's view after being in his or her possession
3. The sample is in the person's possession and is being transferred to a designated secure area
4. The sample has been locked up to prevent tampering after it was in the person's possession.

At no time is it acceptable for samples to be outside of Integral personnel's custody unless the samples have been transferred to a secure area (i.e., locked up). If the samples cannot be placed in a secure area, then an Integral field team member must physically remain with the samples (e.g., at lunch time one team member must remain with the samples).

CHAIN-OF-CUSTODY FORMS

The COC form is critical because it documents sample possession from the time of collection through final disposition. The form also provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

Complete the COC form after each field collection activity and before shipping the samples to the laboratory. Sampling personnel are responsible for the care and custody of the samples until they are shipped. The individuals relinquishing and receiving the samples must sign the

COC form(s), indicating the time and date of the transfer, when transferring possession of the samples.

A COC form consists of three-part carbonless paper with white, yellow, and pink copies. The sampling team leader keeps the pink copy. The white and yellow sheets are placed in a sealed plastic bag and secured inside the top of each transfer container (e.g., cooler). Field staff retain the pink sheet for filing at the Integral project manager's location. Each COC form has a unique four-digit number. This number and the samples on the form must be recorded in the field logbook. Integral also uses computer-generated COC forms. If computer-generated forms are used, then the forms must be printed in triplicate and all three sheets signed so that two sheets can accompany the shipment to the laboratory and one sheet can be retained on file. Alternatively, if sufficient time is available, the computer-generated forms will be printed on three-part carbonless paper.

Record on the COC form the project-assigned sample number and the unique tag number at the bottom of each sample label. The COC form also identifies the sample collection date and time, type of sample, project name, and sampling personnel. In addition, the COC form provides information on the preservative or other sample pretreatment applied in the field and the analyses to be conducted by referencing a list of specific analyses or the statement of work for the laboratory. The COC form is sent to the laboratory along with the sample(s).

PROCEDURES

Use the following guidelines to ensure the integrity of the samples:

1. Sign and date each COC form. Have the person who relinquishes custody of the samples also sign this form.
2. At the end of each sampling day and prior to shipping or storage, make COC entries for all samples. Check the information on the labels and tags against field logbook entries.
3. Do not sign the COC form until the team leader has checked the information for inaccuracies. Make corrections by drawing a single line through any incorrect entry, and then initial and date it. Make revised entries in the space below the entries. After making corrections, mark out any blank lines remaining on the COC form, using single lines that are initialed and dated. This procedure will prevent any unauthorized additions.

   At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date of the transfer. The time the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as FedEx or United Parcel Service (UPS), record the name of the carrier on the COC form. Also enter on the COC form any tracking numbers supplied by the carrier. The time of transfer should be as close to the actual drop-off time as possible. After signing the COC forms and removing the pink copy, seal them inside the transfer container.
5. If errors are found after the shipment has left the custody of sampling personnel, make a corrected version of the forms and send it to all relevant parties. Fix minor errors by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.
6. Provide a COC form and an Archive Record form for any samples that are archived internally at Integral.

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all COC forms to be copied. A discussion of copy distribution is provided in SOP AP-02.

CUSTODY SEAL

As security against unauthorized handling of the samples during shipping, affix two custody seals to each sample cooler. Place the custody seals across the opening of the cooler (front right and back left) prior to shipping. Be sure the seals are properly affixed to the cooler so they cannot be removed during shipping. Additional tape across the seal may be prudent.

SHIPPING AIR BILLS

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), the shipper provides an air bill or receipt. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting the sender's copy of all shipping air bills to be copied at an Integral office. A discussion of copy distribution is provided in SOP AP-02. Note the air bill number (or tracking number) on the applicable COC forms or, alternatively, note the applicable COC form number on the air bill to enable the tracking of samples if a cooler becomes lost.

ACKNOWLEDGMENT OF SAMPLE RECEIPT FORMS

In most cases, when samples are sent to a testing laboratory, an Acknowledgment of Sample Receipt form is faxed to the project QA/QC coordinator the day the samples are received by the laboratory. The person receiving this form is responsible for reviewing it, making sure that the laboratory has received all the samples that were sent, and verifying that the correct analyses were requested. If an error is found, call the laboratory immediately, and document

any decisions made during the telephone conversation, in writing, on the Acknowledgment of Sample Receipt form. In addition, correct the COC form and fax the corrected version to the laboratory.

Submit the Acknowledgment of Sample Receipt form (and any modified COC forms) to be copied. A discussion of copy distribution is provided in SOP AP-02.

ARCHIVE RECORD FORMS

On the rare occasion that samples are archived at an Integral office, it is the responsibility of the project manager to complete an Archive Record form. This form is to be accompanied by a copy of the COC form for the samples, and will be placed in a locked file cabinet. The original COC form remains with the samples in a sealed Ziploc® bag.

STANDARD OPERATING PROCEDURE (SOP) AP-04

SAMPLE LABELING

SCOPE AND APPLICATION

This SOP describes the general Integral procedures for labeling samples, and the three kinds of labels that can be used on a project (i.e., sample labels, sample tags, and internal sample labels). Consult the project-specific sampling and analysis plan (SAP) to determine the exact sample identifiers and sample labels that are required for a given project. If they are not specified in the SAP, then follow the designations below.

SAMPLE IDENTIFIERS

Before field sampling begins, establish sample identifiers to be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., replicates) to ensure proper data analysis and interpretation, 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples, and 3) to track individual sample containers to ensure that the laboratory receives all material associated with a single sample. To accomplish these purposes, each container may have three different codes associated with it: the sample identifier, the sample number, and the sample tag number. These codes and their use are described as follows:

- **Sample Identification Code**—The sample identification code (Sample ID) is a unique designation that identifies where and how the sample was collected. The sample identifier is recorded in the field logbook *only* and is not provided on the sample label or chain-of-custody (COC) form. The sample identifier is a multiple-part code. The first component begins with the letter abbreviation; for example, "SWNS" or "SWNB" to designate the surface water sample was collected from the near-surface or near-bottom of the water column. The second part could identify the sampling event; for example, "1" to designate Round 1 sampling. The third part could contain an abbreviation for whether the station is a single point (SP), a transect (TR), a composite (CO), or a vertically integrated station (VI). The station number would be the final component of the sample identifier. Use leading zeros for stations with numbers below 100 for ease of data management and correct data sorting.

  If appropriate, add a supplemental component to the sample identifier to code field

duplicate samples and splits. Use a single letter (i.e., a suffix of "A" and "B") to indicate field duplicates or splits in the final component of the sample identifiers. For equipment decontamination blanks, assign sequential numbers starting at 900 instead of station numbers. Use a sample type code that corresponds to the sample type for which the decontamination blank was collected. Additional codes may be adopted, if necessary, to reflect sampling equipment requirements (see project-specific SAP).

Examples of sample IDs are as follows:

- SWNS-1-SP-002: Surface water sample collected from the near-surface at a single point during Round 1 from Station 2.
- SWNB-1-TR-010-A: Duplicate surface water sample from the near-bottom transect during Round 1 from Station 10.

- **Sample Number**—The sample number is an arbitrary number assigned to each distinct sample or split that is shipped to the laboratory for separate analysis. The sample number appears on the sample containers and the COC forms. Each sample will be assigned a unique sample number. All aliquots of a composited field sample will have the same sample number. In cases where samples consist of multiple bottles from the same location, assign each bottle the same sample number and time. However, assign replicates from the same location different sample numbers and times. Sample numbers of related field replicates will not necessarily have any shared content.

  Each field split of a single sample will also have a different sample number and time. The sample number is generally a unique six-digit number that includes a two-digit media code and a four-digit number. The media code may be specific to the sampling project, but the Integral default codes are as follows:

  - SS—Surface soil
  - BH—Subsurface soil or rock (typically from borehole)
  - GW—Groundwater
  - SW—Surface water
  - PW—Pore water
  - SD—Sediment
  - BT—Biota or biological tissue

    The exact sample numbering scheme may vary from project to project. Variances in the sample numbering scheme will be described in the project-specific SAP for the field event. Example sample numbers are PW0001, PW0002, PW0003, etc.

- **Tag Number—**Attach a different tag number to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, assign each container the same sample number and a different sample tag. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted).

  The sample tag number is a unique five- or six-digit number assigned to each sample label (or "tag") for multiple bottles per sample. Integral sample labels come with a preprinted sample tag number. The tag number provides a unique tracking number to a specific sample bottle. This allows for greater flexibility in tracking sample bottles and assists in field quality control when filling out documentation and shipping. Sample tags are not used by many other consultants, and there may be resistance from such firms during teaming situations. However, experience has shown that tags can be very valuable, both in the field and while processing data from field efforts.

  Record tag numbers on the COC form. Laboratories use tag numbers only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Assign sample numbers sequentially in the field; sample labels are preprinted with sequential tag numbers.

SAMPLE LABELS

Integral sample labels are designed to uniquely identify each individual sample container that is collected during a sampling event. Field sampling teams are provided with preprinted sample labels, which must be affixed to each sample container used. Fill out the labels at the time the samples are collected, documenting the following information:

- Sample number
- Study Area name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- A unique number (commonly referred to as the "Tag Number") that is preprinted on the label consisting of five or six digits; used to identify individual containers.

SAMPLE TAGS

Integral sample tags are designed to be affixed to each container that is used for a sample. Sample tags are required only for environmental samples collected in certain U.S.

Environmental Protection Agency (EPA) regions (e.g., EPA Region 5). Field crews are provided with preprinted sample tags. Attach sample tags to each individual sample container with a rubber band or wire through a reinforced hole in the tag. Mark all sample tag entries with indelible ink. Fill out the tags at the time the samples are collected, documenting the following information:

- Sample number
- Study Area name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- Type of analysis.

A space for the laboratory sample number (provided by the laboratory at log-in) will also be provided on the sample tag.

INTERNAL SAMPLE LABELS

For benthic infaunal samples, wash away the sediment from the sample and collect the remaining benthic infauna into a sample container. Affix sample label (as discussed above) to the outside of the sample container. In addition, place an internal sample label inside the sample container. This internal sample label is made of waterproof paper; be sure to make all internal sample label entries with pencil. Fill out the internal sample labels at the time the samples are collected, documenting the following information:

- Sample number
- Study Area name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservative used (e.g., formalin).

STANDARD OPERATING PROCEDURE (SOP) AP-05

CHARACTERIZATION-DERIVED WASTE HANDLING

SCOPE AND APPLICATION

This SOP presents the method to be used for handling wastes generated during field sampling activities that could be hazardous. These wastes are referred to as characterization-derived waste and are subject to specific regulations.

All disposable materials used for sample collection and processing, such as paper towels and gloves, are not considered characterization-derived wastes and will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies will be removed from OU4 by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill.

EQUIPMENT AND REAGENTS REQUIRED

- 55-gallon drums (or appropriately sized waste container)
- Paint markers
- Tools (to open and close drum)
- Ziploc® bags
- Drum labels.

PROCEDURES

1. Place solid wastes that need to be containerized in properly labeled, DOT- approved, 55-gallon drums.
2. Properly close, seal, label, and stage all filled or partially filled drums before demobilization. Properly profile full drums and have them shipped off OU4 to a RCRA Subtitle C facility.

3. Sampling activities generate personal protective equipment and miscellaneous debris that require disposal. Remove gross contamination from these items, and place the items in plastic bags. It is acceptable to store these items in plastic bags as an interim measure. At the end of each day, dispose of the bags at an appropriate solid waste facility dumpster.

STANDARD OPERATING PROCEDURE (SOP) SD-01

DECONTAMINATION OF SEDIMENT SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either inorganic or organic materials. To prevent potential cross contamination of samples, all reusable sediment sampling and processing equipment is decontaminated before each use. At the sample collection location, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All sediment sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the Investigation Area-specific health and safety plan (HASP).

Sampling equipment (e.g., van Veen, Ekman, Ponar, core tubes) may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment used for both analyte groups should follow the order of a detergent wash, rinse with water from the Investigation Area, organic solvent rinses, and final rinse with water from the Investigation Area. Sample processing equipment (e.g., bowls, spoons) has a final rinse with distilled/deionized water rinse instead of water from the Investigation Area. If the surface of stainless steel equipment appears to be rusting (possibly due to prolonged contact with organic-rich sediment), it should undergo an acid rinse and a rinse with water from the Investigation Area at the end of each sampling day to minimize corrosion.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following:

- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or water from the Investigation Area
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)

- Properly labeled squirt bottles
- Funnels
- Alconox®, Liquinox®, or equivalent industrial detergent
- Pesticide-grade acetone and hexane (consult the project-specific field sampling plan [FSP], as the solvents may vary by EPA region or state)
- 10 percent (v/v) nitric acid (reagent grade) for inorganic contaminants
- Baking soda
- Long-handled, hard-bristle brushes
- Extension arm for cleaning core liners
- Plastic sheeting, garbage bags, and aluminum foil
- Core liner caps or plastic wrap and rubber bands
- Personal protective equipment as specified in the health and safety plan.

PROCEDURES

Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., < 1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not an analyte in the samples. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is sometimes slightly more effective than other solvents, its use is discouraged due to potential toxicity to sampling personnel.

The specific procedures for decontaminating sediment sampling equipment and sediment compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap water or water from the Investigation Area to remove visible sediment. Perform this step on location for all equipment, including core liners that will not be used again until the next day of sampling. After removing visible solids, set aside sampling equipment that does not need to be used again that day; this equipment should be thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap water or water from the Investigation Area. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double rinse the equipment with tap water or water from the Investigation Area and set right-side-up on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate[1] the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse equipment with tap water or water from the Investigation Area and set right-side-up on a stable surface to drain thoroughly.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). Hold core liners over the waste container and turn them slowly so the stream of solvent contacts the entire surface. Turn the sample apparatus (e.g., grab sampler) on its side and open it to wash it most effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.

[1] Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.

7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container (which may need to be equipped with a funnel). If necessary, widen the opening of the squirt bottle to allow enough solvent to run through the core liners without evaporating. (Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the equipment was not thoroughly rinsed with acetone or that the acetone that was purchased was not free of water.) When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the acetone and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.
8. Do a final rinse with water from the Investigation Area for the sampling equipment (i.e., van Veen, Ekman, Ponar, core tubes) and with distilled/deionized water for processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

   If the sample collection or processing equipment is cleaned at the field laboratory and transported to the Investigation Area, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.
10. Rinse or wipe with a wetted paper towel all stainless-steel equipment at the end of each sampling day with 10 percent (v/v) normal nitric acid solution. Follow with a freshwater rinse (water from the Investigation Area is okay as long as it is not brackish or salt water).
11. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles and disposed of at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Metals and Conventional Parameters Only

The specific procedures for decontaminating sediment sampling equipment and sediment processing equipment are as follows:

1. Rinse the equipment thoroughly with tap water or water from the Investigation Area to remove the visible sediment. Perform this step on location for all equipment, including core liners that will not be used again until the next day of sampling. Set aside pieces that do not need to be used again that day; these pieces should be and thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap water or water from the Investigation Area. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double-rinse the equipment with tap water or water from the Investigation Area and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate[2] the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse sampling equipment with tap water or water from the Investigation Area and set right-side-up on a stable surface to drain. Double-rinse processing equipment with distilled/deionized water and allow to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

[2] Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.

If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the Investigation Area, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.

7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SD-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SEDIMENTS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates, equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material; for the purposes herein, all types of reference materials are referred to as standard reference material, or SRM) for sediment sampling efforts. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples are an equipment rinsate blank, a field duplicate, and trip blanks if samples are to be analyzed for volatile organic compounds (VOCs). Definitions of all potential quality control samples are described below.

As part of the quality assurance/quality control (QA/QC) program, all field quality control samples will be sent to the laboratories "blind." To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers with preservatives that are required to complete the field quality control sample for the applicable analyte list shall be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should be recorded only in the field logbook. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent such an occurrence, regular samples should be selected and marked on the chain-of-custody/sampling analysis request (COC/SAR) form or the laboratory should be instructed to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Field quality control samples will be prepared at least once per sampling event, and certain types will be prepared more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of

field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality control sample per 20 is indicated and 28 samples are collected, 2 quality control samples will be prepared. Field quality control samples for sediment sampling activities should be prepared consistent with the requirements discussed below and at the frequency indicated unless different frequency requirements are listed in the FSP and QAPP.

The following table lists the quality control sample types and suggested frequencies for sediment sampling programs. Because sediment quality control sampling may require assessment of cross-contamination, additional blanks may be required. A detailed explanation of each quality control sample type with the required preparation follows.

Table 1. Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling location	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling location	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling location	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples.
Equipment rinsate blank	ER	Sampling location	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter wipe	FW	Sampling location	Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be wiped over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter paper blank	FB	Sampling location	Clean, unused Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be sent to the analytical laboratory	Minimum of one for each lot number of filter papers used.
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type.
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment.

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler.
Environmental blank	EB	Field	Bottle filled at sample location with deionized water	One per 20 samples.
Standard reference material	SRM	Field laboratory or sampling location	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode.

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Field duplicate (or split) samples are collected to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Field duplicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of field duplicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Field replicates will be prepared by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Field replicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The actual number of field replicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The matrix spike/matrix spike duplicate (MS/MSD) analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated sediment stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of extra bottles collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Equipment rinsate blanks will be used to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Equipment rinsate blanks will be prepared by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, and then transferring the water to the appropriate sample containers and adding any necessary preservatives. Equipment rinsate blanks will be prepared for all inorganic, organic, and conventional analytes at least once per sampling event per the type of sampling equipment used. The actual number of equipment rinsate blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

FILTER WIPES

Filter wipe samples will be used to help identify possible contamination from the sampling environment or from the decontaminated sediment sampling equipment (e.g., sediment grab sampler, stainless-steel bowls and spoons, shovel, trowel).

Filter wipe samples will be prepared by grasping a piece of clean, ashless filter wipe/paper with decontaminated tongs and/or tweezers and wiping down all surfaces of dry, decontaminated equipment that comes into contact with the sediment sample (e.g., stainless-steel spoon, inside of sediment grab sampler). Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter wipes/papers will be from the same lot used to prepare the filter paper blanks (see below), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be used for each equipment type, solid matrix type, and analysis type. For example, if two pieces of equipment were used for sediment sampling (trowel and stainless-steel spoon) and both metals and

organic compounds were being analyzed, then a total of four filter wipes/papers would be sent to the analytical laboratory.

Tongs and/or tweezers will be used to handle the filter wipe/paper, and all sediment sample-exposed surfaces will be thoroughly wiped down using one piece of filter wipe/paper (per equipment type and for each analysis). The filter wipe sample will then be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory. The filter wipe/paper box will be stored in a clean glass container and must NOT be stored in a plastic bag. In moist environments, the filters should be wrapped thoroughly in aluminum foil to protect them from moisture.

Filter wipe samples will be prepared for all inorganic and organic analytes at least once per sampling event per the type of sampling equipment used. The actual number of filter wipe samples prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter wipe sample collection may vary by EPA region or state).

FILTER PAPER BLANKS

Whenever a filter wipe sample is prepared in the field, a filter paper blank will also be prepared in the field to evaluate potential background concentrations present in the filter paper used for the equipment filter wipe sample.

Filter paper blanks will be prepared by using tongs and/or tweezers to remove the clean ashless filter paper from its box. Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter papers will be from the same lot used to prepare the filter wipe samples (see above), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be sent to the analytical laboratory for each type of analysis to be performed (i.e., inorganic or organic analytes). The filter paper blank will be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory.

Filter paper blanks will be collected at a minimum frequency of one for each filter lot number. The actual number of filter paper blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter paper blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Bottle blanks are submitted along with sediment samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, one bottle

blank per lot of prepared bottles will be submitted for analysis. If more than one type of bottle will be used in the sampling (e.g., high-density polyethylene or glass), then a bottle blank should be submitted for each type of bottle and preservative. The actual number of bottle blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC/SAR form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Trip blanks will be used to help identify whether contaminants may have been introduced during the shipment of the sediment samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/ deionized water into two 40-mL VOC vials and tightly closing the lids. Each vial will be inverted and tapped lightly to ensure no air bubbles exist.

The trip blanks will be transported unopened to and from the field in the cooler with the VOC samples. A trip blank is labeled and placed inside the cooler that contains newly collected VOC samples and it remains in the cooler at all times. A trip blank must accompany samples at all times in the field. One trip blank (consisting of a pair of VOC vials) will be sent with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

Temperature blanks will be used by the laboratory to verify the temperature of the samples upon receipt at the testing laboratory. Temperature blanks will be prepared at the testing laboratory by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank shall be included with each sample cooler shipped to the testing laboratory.

ENVIRONMENTAL BLANKS

The environmental (field) blank is prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If unpreserved bottles are to be used, then the appropriate preservative (i.e., for metals samples use a 10 percent nitric acid solution to bring sample pH

to 2 or less) must be added, as may be required. Environmental blanks should be collected at a minimum frequency of 1 in 20 samples. The actual number of environmental blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection location, fill the sample bottle with distilled/deionized water, and then seal it. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

SRMs are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. The SRMs have undergone multi-laboratory analyses using a standard method that provides certified concentrations. When available for a specific analyte, SRMs provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several SRMs may be required to cover all analytical parameters. For all analytes where available, one SRM will be analyzed at a frequency of one per 50 samples. The actual number of SRMs analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of SRM analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SD-08

SEDIMENT CORE COLLECTION USING A VIBRACORER

SCOPE AND APPLICATION

This SOP describes the procedure for collecting and processing sediment core samples using a vibracore system, which collects continuous and relatively undisturbed sediment cores. This method of sediment coring is performed from a boat and uses high-frequency low-amplitude vibration to break down the frictional resistance of the sediment and allow the core tube to penetrate into the sediment with minimal distortion. It is best used for sampling coarse, consolidated sediment and very cohesive sediment, where static weight (e.g., piston-type or conventional gravity corers) will not produce adequate penetration into the sediment. In addition, the vibracorer offers a high rate of production, superior retention of shallow samples, and a greater sample volume compared to conventional drilling equipment.

Vibracorers generally consist of a metal corer barrel (usually a 4-in.-outside-diameter, aluminum core barrel) with a location-dedicated polycarbonate or Lexan®-lined core tube, and a vibrator mechanism attached to the top of the barrel. The vibration is created either by an electric motor, a hydraulic system, or a pneumatic piston attached to the top of the barrel. Therefore, a generator or air compressor is needed on board to power the corer. The pneumatic piston does not have the same function as a piston in a piston corer. Because vibracorers generally do not have a piston in the corer, some compaction and/or bypass will occur, and recovery will be less than 100 percent.

A continuous sediment sample is retained within the tubing with the aid of a stainless-steel core cutter/catcher or nosecone attached to the bottom of each aluminum tube.

It is always best to keep the core in a vertical position to prevent the top layers of sediment (i.e., the top 5 to 15 cm) from slumping. However, in many cases, it is not feasible to process the core in a vertical position because the tripod needs to be at least twice the height of corer, and sectioning and logging the sample would have to be performed from a ladder. For studies that specify sectioning the sample into coarse intervals (>20 cm), processing the core in a horizontal position will generally not significantly disrupt the stratigraphy. For studies that specify shorter intervals (<5–10 cm), processing the core in a horizontal position is likely to disrupt stratigraphy. In this case, the top layers of sediment that have high water content should be sectioned while the core is in a vertical position, and when the sediment becomes thicker, the corer can be laid horizontally.

PROCEDURES

Decontamination

To prevent potential cross-contamination of samples, all reusable sediment sampling equipment must be decontaminated prior to use at each station and between field replicates.

Before each station is sampled, decontaminate the inner surfaces of the corer or core tube liner and all stainless-steel sample compositing equipment. Prior to sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox®), rinsed with site water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination. Details on correct decontamination procedures can be found in SOP SD-01, *Decontamination of Equipment—Sediment*. The project-specific field sampling plan (FSP) should also be consulted to determine any project-specific decontamination procedures. The personnel performing the decontamination procedures will wear protective clothing as specified in the site-specific health and safety plan.

All solvent rinsates (if used) will be collected into a bucket or tub and allowed to evaporate over the course of the day. Any rinsate that has not evaporated by the end of the sampling event will be containerized and disposed of in accordance with applicable regulations.

Vibracorer Deployment and Retrieval

The following procedures are based on using the vibracorer aboard a boat equipped with a tripod or A-frame of sufficient height to allow recovery of the core (see project-specific FSP for information on target coring depth), and a power winch. On pontoon boats, the tripod is centered over a hole in the floor, whereas on other boats, the corer may be lowered over the side or stern. To obtain cores of high quality, the boat must be anchored with at least three anchors so the boat will not drift during the coring process.

1. Maneuver the sampling vessel to the targeted sampling location using the positioning procedures and minimum water depth restrictions.
2. Deploy 3- or 4-point anchor system to maintain position; record and monitor position throughout core acquisition.
3. Once on location, measure the water depth (depth to top of sediment) using the onboard depth sounder (fathometer) or lead line and record measured depth in the field logbook. If the water level is affected by tides, obtain tide level measurements and calculate tidal height in feet above mean low water. The date, time, weather, and water conditions (e.g., high wave activity, strong currents, turbidity, tidal flux) should also be recorded in the field logbook.

4. Assemble the decontaminated core tube, liner, core catcher, and cutter heads (or nose cone depending on the model of vibracorer used), using care to not contact decontaminated surfaces. Attach assembled vibracorer to winch cable. Note that several decontaminated catchers and cutterheads will be on hand, in case of loss. Core catchers and cutter heads can be decontaminated and reused for subsequent core collection.
5. Attach a tape measure to the vibracorer or mark the winch cable in 1/2-ft increments to measure penetration depth.
6. Inspect connections of winch cable and electrical or pneumatic lines to confirm they are secure.
7. Signal the winch operator to slowly raise the vibracorer into a vertical position and guide the vibracorer (with core liner, valve, core catcher, and cutterhead in place) overboard until it is clear of the vessel.
8. Using the winch, slowly lower the vibracorer through the water column at a speed of about 1 ft/s to avoid creating a bow wake or overturning of the vibracore. Stop lowering the corer a few feet above the sediment and confirm that the boat has not drifted.
9. Continue lowering the vibracorer until the tip of the core is resting on the sediment or to the depth recorded by the fathometer, depending on the consistency of the sediment. Record the vibracorer depth as derived from the attached tape measure or marked winch cable. Measurements will serve as a basis for determining penetration depth.
10. Resume lowering the corer at about 1 ft/s. When the nosecone or core catcher contacts the sediment, turn on the vibracorer motor. The vibracorer is then allowed to slowly penetrate the sediments. Initially, light tension should be maintained on the cable to keep the corer from tipping over.
11. Lower the vibracorer to the target penetration depth as measured by the attached tape measure or marked winch cable. If the targeted penetration depth is met, proceed to the next step; if refusal is met, retrieve the vibracorer, perform gross decontamination (i.e., rinse with river water and brush off visible sediment from the outside of the core barrel) and re-attempt at new location offset at least 3–5 ft from original location.
12. When the target penetration depth is reached, or refusal occurs, turn off the vibracorer and record the time, penetration depth, angle of the cable relative to the boat, and any other observations.
13. Slowly withdraw the core from the bottom sediments at a constant rate, to keep it upright and not dislodge any sediment from within the core barrel, and raise it to the surface.

14. With the corer hanging in a vertical position, clean the vibracorer assembly by hosing down the equipment with site water prior to bringing the core onboard the sampling vessel. If the corer is not plugged, care should be taken not to direct water into the open end of the core barrel.
15. Slowly guide the core onboard the vessel; use care to avoid jostling that might disturb the integrity of the core. Care must be taken to keep the top end of core elevated to prevent sediment from "pouring" out. Use a sawhorse or equivalent to elevate the top of the core. As soon as the nosecone clears the water surface, the bottom of the corer may be plugged with a rubber stopper to prevent loss of sediment.
16. Before removing the core tube from the vibracorer, visually inspect the nosecone or core cutter/catcher to ensure that proper penetration has been attained and that there is no obvious loss of sediment from the tube. Record any presence of noticeable odors, the core penetration depth, and physical characteristics (e.g., color, texture, odor) of the sediment sample as observed at the ends of the tube in the field logbook or on the Field Sediment Core form. In addition, note any sheen in the water in the field logbook.
17. If the core will be processed horizontally, slowly lay the corer down. Unscrew the cutter head (or nosecone) and carefully remove the core catcher, while retaining as much sediment as possible.
18. While removing the core catcher (or nosecone), be ready to immediately seal the end of the core liner by placing clean aluminum foil and a plastic cap over the open end.
19. Carefully remove the core liner that contains the sample by lifting the lower end from the deck as needed to provide clearance. Affix core cap, wrap with tape, label core liner and end of core, remove valve from top of core liner, stand core upright, and place in a processing rack or tray to allow the sediment at the top of the core to settle. Avoid sudden movements to the core that would disrupt the sediment interface.
20. While waiting for sediment to settle, prepare the Field Sediment Core form. Identify any debris and note its depth in the core and what the debris is, if possible.
21. Once resuspended sediment has settled, measure the length of the recovered core, calculate percent recovery (100 x recovered length/penetration depth), and record in the logbook or on the Field Sediment Core form.
22. Check the core for acceptability. The following acceptability criteria should be satisfied:
    - The core tube is not overfilled with sample so that the sediment surface presses against the bottom of the vibracorer head.
    - Overlying water is present (indicates minimal leakage).
    - The overlying water is not excessively turbid (indicates minimal disturbance).

- The desired penetration depth (see project-specific FSP for required penetration depth) or refusal has been reached.

Depending on requirements of the project-specific FSP, a core may be rejected based on percent recovery. Commonly, a core is deemed unacceptable if recovery is less than 80 percent. If recovery is less than 80 percent, the core sample will be retained for possible processing, while additional sampling attempts are made to collect a core with greater than 80 percent recovery. If subsequent attempts result in recoveries of less than 80 percent, then the sample with the highest percent recovery may be used for analysis. The number of attempts to collect an acceptable sample will be specified in the project-specific FSP. If recovery is less than 80 percent, the core may be acceptable if the penetration depth is deeper than the target core length. In this case, the recovered length should be equal to the target length.

23. Once sufficient time has been allowed for the sediment to settle (i.e., no sediment is suspended in the overlying water), use a decontaminated saw to cut a drain-slit or a decontaminated drill bit to drill in the side of the core liner approximately 1 to 2 in. above the sediment–water interface; allow excess water to drain. Cut excess polycarbonate liner with decontaminated blade and use a siphon to decant off the overlying water. Ensure that the saw blade, drill bit, or siphon does not contact the sediments and that fine-grained suspended sediment is not removed.
24. Cut cores into manageable sections (3–4 ft) aboard the vessel immediately after their retrieval. Cap each section with aluminum foil and plastic caps, and seal with duct tape. Mark the core with permanent marker using a unique number or alphanumeric code identifying sampling location, core number, core section, and segment orientation (i.e., which end is up). Following sectioning, store the cores in an upright position onboard the vessel in a core box and have them transported periodically throughout each field day by small boat to a field processing area where they are to be stored upright under custody on ice or refrigerated at 4°C to await processing.
25. In preparation for next core, thoroughly rinse the interior of the core barrel until all loose sediment has been washed off. Repeat process at next sampling location. Continue coring until requirements are met.

In situations where there is significant surface water depth and/or water current that could cause the vibracorer setup to lean at an unacceptable angle, a buoyant frame or rigid frame configuration should be used.

With the buoyant frame, the vibracorer is maintained in proper vertical position by two guidelines held taut between a float package and a weight stand. The larger weight stand is provided with ballast boxes so that easy-to-find ballasting material such as lead bags or scrap metal can be used in the field. For deployment, the vibracorer is lowered with the weight

stand hanging on its guidelines from the vibrahead. The float package is hooked up to the guidelines when the vibrahead reaches the deck level.

After coring and pull-up, the system is retrieved in the reverse manner. In case of limited deck space or overhead clearance, or to further accelerate the procedure on the water, the weight stand can be left in as overboard cradle.

Sample Handling, Storage, and Processing

Cores should be processed concurrently with core collection, and every effort should be made to ensure cores are processed within 24 hours of collection. Cores awaiting processing will be sealed tightly at both ends and stored upright in ice or in a refrigerator. If core collection outpaces processing such that significant delays in core processing appear likely, core collection will be suspended to allow the core processing to catch up.

As mentioned above, once coring has been completed at a given location, the cores will be transported in an upright position on ice to a designated field processing area, where they will be logged and processed. The field processing area will be equipped with a core-cutting table, core-processing tables, a decontamination area, and a storage area with appropriate refrigeration. Appropriate lighting will be installed in the field processing area so that consistent, high quality photographs can be taken of the opened cores. Care should be taken to create a field processing area that minimizes the potential for outside contamination.

Sample processing includes removing the sample from the liner, recording observations of sample characteristics, mixing subsamples, and distributing the sample to containers for shipping to the testing laboratory. Vibracore processing most often consists of the following steps:

1. Cut each core tube along the long axis using decontaminated hook blade. Rotate the tube 180^{o} and cut again.
2. After each core is cut, move the entire core tube to an aluminum foil-covered table and open it so that it can be systematically logged, described, and photographed.

However, depending on the project-specific FSP, the core may be extruded from the liner and cut into the specified intervals as it emerges or the core liner may be cut into sections, sealed, and shipped intact to the testing laboratory.

Core Observations

1. Verify that the length of the core, water depth, and all required position data have been recorded in the field logbook together with all pertinent observations and communications with the field team leader.
2. After each core is cut open, describe the sediment on a Field Sediment Core form in the field processing area notebook. When recording the information for each core, follow the guidelines below:

- Physical sediment description (i.e., sediment type [e.g., silt, sand], density/consistency, color) (see SOP SL-04, Field Classification of Soil, based on ASTM D 2488-00 [ASTM 2000])
- Odor (e.g., hydrogen sulfide, petroleum, creosote)
- Visual stratification, laminations, and lenses
- Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
- Moisture content
- Vegetation
- Approximate percentage of vegetation
- Debris
- Approximate percentage of debris
- Presence of biological structures (e.g., detritus, shells, tubes, bioturbation, live or dead organisms, chironomids)
- Approximate percentage of biological structures
- Presence of a sheen
- Other distinguishing characteristics or features.

3. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals (check project-specific FSP for sample interval definition; depending upon the project-specific requirements the sample interval could be based on lithology or it could be set to a specific interval [e.g., 1 ft]).
4. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).
5. Photograph the cores after they have been described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs may be used later in the production of digital core logs.

Mixing and Sample Preparation

1. After the sample is characterized and the core observation logged on the Field Sediment Core form, remove the specified sample interval using a stainless-steel spatula or spoon (see project-specific FSP for correct sampling interval). Exercise care to not include sediment that is in direct contact with the core tube. With the approval of the field team leader, and using a decontaminated stainless-steel instrument, carefully remove unrepresentative material (e.g., large shells, stones). Exercise care not to touch the sediment during this process. Note any unrepresentative material removed from the sample in the field processing area notebook.
2. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid-volatile sulfides), and place them directly into sample containers without homogenization. Completely fill the sample container so that there is no headspace or entrapped bubbles.
3. Transfer the remainder of the sample interval to a decontaminated stainless-steel bowl for homogenization. If additional sediment volume is required to fill all sample bottles (see project-specific FSP) and multiple cores need to be collected at a given station, cover the compositing bowl covered with aluminum foil (dull side down) to prevent sample contamination (e.g., from precipitation, engine exhaust, splashing water) and place in a cool dark place until the next core from that location is processed.
4. After all the sediment is transferred to the compositing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
5. Distribute subsamples to the various containers specified in the project-specific FSP and preserve the samples as specified in the project-specific FSP. Briefly stir the sediment in the compositing bowl between each spoon transfer to the sample containers.
6. Subsequent intervals should be processed in the same way.

Field Quality Control Samples

If additional volumes of sediment are required to perform all analyses including quality control analyses, an additional core may need to be collected from the same location and subsampled and homogenized accordingly. Details on collection of field quality control samples (e.g., field duplicates) will be specified in the project-specific FSP. Details on collection of field quality control samples and preparation of the certified reference materials can be found in SOP SD-02, *Preparation of Field Quality Control Samples—Sediment,* and SOP SD-03, *Preparation of Reference Materials—Sediment*. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality

control samples will be described in the project-specific FSP and quality assurance project plan.

Field Measurements

A water depth measurement must be collected at every sampling location. Depending on the specific project objectives, it may be necessary to perform field measurements of the *in situ* environment. Possible field measurements include temperature and pH of the sediment at the sediment-water interface and concentration of dissolved oxygen, salinity, or conductivity in the overlying water. Details on collection of field measurements can be found in SOP SD-11, *Field Analyses for Sediment*. The specific field measurements, if any, will be specified in the project-specific FSP.

Station Location Coordinates

Station locations for all field sampling will be determined using a differential global positioning system (DGPS) or by surveying. The accuracy to which the latitude and longitude of a station location is determined will be specified in the FSP. At a minimum, a DGPS capable of providing latitude and longitude coordinates with an accuracy of approximately 3 m is recommended. The DGPS consists of two satellite receivers linked to each other by a VHF telemetry radio system. The receiver will be on the sampling vessel. Details on collection of very accurate station coordinates can be found in SOP AP-06, *Navigation.*

Sample Custody and Shipping

Sample custody will be maintained in accordance with procedures outlined in SOP AP-03, *Sample Custody*. All samples will be packaged and shipped with other samples in accordance with procedures outlined in SOP AP-01, *Sample Packaging and Shipping*.

Troubleshooting

Insufficient Sample

The corer may not collect enough sediment because of 1) inadequate penetration, 2) adequate penetration but poor recovery due to compaction, 3) adequate penetration but poor recovery as a result of bypass, or 4) adequate penetration but loss of sample during retrieval. Compaction and bypass are two different artifacts that are difficult to distinguish and quantify. Following is an approach to identifying the causes and remedies of insufficient sample length. Keep in mind that a combination of these causes may occur:

- **Inadequate Penetration**—Allow more vibration time at the refusal depth, or increase the vibrator frequency.

- **Poor Recovery Due to Compaction**—Compaction is the process of rearranging the sediment particles, so that less volume is occupied by pore water, which results in a shorter column of sediment in the corer than *in situ*. Compaction occurs only in clean coarse silt, sand, and gravel sediments that have a high hydraulic conductivity and are not terminally compacted *in situ*. Fine-grained cohesive sediment (i.e., low hydraulic conductivity) does not compact. The key feature of compaction is that all of the solids ahead of the nosecone are collected as the corer penetrates. So, although the calculated recovery is less than 10 percent, 100 percent of the sediment solids were recovered. Therefore, if the sample has poor recovery, is composed of clean coarse-grained materials, and there is no evidence of sediment falling out the bottom, then the sample is likely to have been compacted. Depending on the project-specific FSP, the specified sample intervals may be shortened proportional to recovery. Because compaction of the solids displaces pore water, minimal compaction is needed for cores that are intended for porewater studies, or cores that will be analyzed for substances that have low K_d values. Vibration in vibracorers is known to rearrange particles, which leads to compaction, so another type of corer may be appropriate if compaction is a problem.
- **Poor Recovery as a Result of Bypass**—Bypass is the process of pushing sediment out of the path of the nosecone/corer as it penetrates the sediment. This is caused by the friction of sediment inside the core liner making it difficult for more sediment to enter the tube. This is most pronounced in fine-grained sediments that have low hydraulic conductivity, or layers of hard and soft sediment, or long cores. The low hydraulic conductivity prevents porewater from being displaced, so compaction cannot occur. Fine-grained sediments in this context are those in which particles cannot be felt between the thumb and forefinger of an ungloved hand. These are generally "sticky" or cohesive sediments. Therefore, if a sample has poor recovery, is fine grained and cohesive, and there is no evidence of sediment falling out the bottom, then some of the sediment column has likely been bypassed.
- **Poor Recovery Because of Loss of Sample during Retrieval**—This is often diagnosed by observing some of the core falling out the bottom as the corer approaches the water surface during retrieval, or a core liner that is empty near the bottom. Sample slipping out the bottom of the corer can be caused by a loss of suction or noncohesive sediment that does not stick to the liner wall. Depending on the specific design of the vibracorer, there are several places at which suction can be lost. These may include the valve seat, the valve assembly, the nose piece, and couplings between the barrel and extensions. To prevent loss of suction, Teflon® plumber's tape should be used on all the threaded connections, and the valve assembly should be clean. For coarse-grained sediment (e.g., clean coarse sand and gravel, and shells) that is non-cohesive and falls out the bottom of the corer, it is sometimes possible to penetrate to a lower layer that is finer grained and will effectively plug the bottom of the core. As mentioned above, core catchers may are used to retain sediment in a vibracorer, although they should not be

used if the surface sediments have high water contents and are to be sectioned at less than about 2 inch intervals.

Because recovery can be an important indicator of corer performance, sediment characteristics, and sample quality, some simple tests can be performed as a diagnostic tool. Penetration of the corer can be measured by putting Velcro® tape on the outside of the corer. Velcro® tape can also be used on the inside of the liner during testing to see how far up inside the liner the sediment interface moves, how much sample slips out the bottom, and how much compaction or bypass occurs.

Notes

1. For long cores that require more than one piece of liner, squarely cut the ends of both pieces with a plastic pipe cutter, butt the ends of the two pieces of liner squarely together and tape them securely so no leaks occur. Do not use too many layers of tape or the liner will not fit into the barrel. Do not use duct tape for this process. Use a high quality tape (i.e., 3M 3750) and dry the tubes before applying.
2. Sometimes tripods are not tall enough to lift the corer so that the barrel will clear the top edge of the liner when removing the liner. To remove the liner in this case, upon unscrewing the cutter head (or nose piece), lower the cutter head (or nose piece) and liner into a pail that has a rope securely tied to the handle. While the corer is raised by the winch, lower the pail through the hole in the deck and into the water (if necessary) until the top edge of the liner clears the bottom edge of the barrel. Then lift it back onto the deck.
3. If the vibracorer does not penetrate significantly or if the cable is let out too quickly, the vibracorer will contact the bottom, tip over, and fall sideways. When this happens, the line will initially go slack, then quickly snap to the side and take up the slack. In this case, reject the core and begin again.
4. A good measure of whether the vibracorer collected the sediment-water interface is to inspect the interface for a thin layer (about 1 mm) of olive green benthic or detrital algae. Also, if the core liner is rotated back and forth gently, the top centimeter will appear to have a gelatinous response.
5. It is sometimes impossible to collect an intact interface because gas bubbles are commonly released from sediment when the corer contacts the sediment. The released gas bubbles entrain surface sediment and cause the overlying water to become turbid. If this is the case, gas bubbles in the sediment can likely be observed through the liner wall.

REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

STANDARD OPERATING PROCEDURE (SOP) SD-11

FIELD ANALYSES FOR SEDIMENTS

SCOPE AND APPLICATION

Several physical and chemical sediment parameters are best measured in the field because of the unstable nature of the parameter, or because the information is needed to direct further sampling. Four sediment field parameter measurements are described in this SOP: percent fines, pH, redox potential, and interstitial salinity.

PERCENT FINES

This procedure provides a gross field measurement of percent fines in a sediment sample. This field measurement is not intended to take the place of grain size distribution analysis in the laboratory, but to aid in directing collection of toxicity test samples and reference samples, which can be dependent upon percent fines.

Equipment and Reagents Required

Equipment required to perform this field measurement includes:

- USA Standard Testing Sieve #230 (63 μm opening)
- 50-mL measuring cup
- 100-mL graduated cylinder
- Small plastic funnel
- Teaspoon
- Squirt bottle filled with water.

Procedures

Once a sediment sample has been collected, carry out the following procedures:

1. Thoroughly rinse the sieve and all other equipment and visually inspect to ensure that no sediment or other detritus is present.

2. Collect a sediment aliquot from the grab sampler in the 50-mL cup, ensuring that exactly 50 mL is collected by "shaving" excess sediment from the top of the cup and rinsing any sediment off the sides of the cup.
3. Transfer the sediment aliquot from the 50-mL cup to the sieve using the spoon. Thoroughly rinse the cup and the spoon into the sieve with water to ensure that the entire aliquot has been transferred.
4. Gently rinse the sieve with running water and observe the stream of water coming from the bottom of the sieve. During this step, the fines are being rinsed away. Rinse until the stream of water appears clear, which indicates that all fines have passed through the sieve. Gently rinse the remaining sediment to one side of the sieve.
5. Place the plastic funnel into the 100-mL graduated cylinder and position the lip of the sieve over the funnel. Using the squirt bottle, rinse the sediment into the graduated cylinder, directing the stream of water through the back of the sieve. Continue rinsing until all sediment has been transferred to the graduated cylinder. If needed, rinse any sediment that may have adhered to the funnel. The rinse water should not overflow the graduated cylinder. If it appears that the graduated cylinder will overflow before all sediment has been transferred, either discard the sample and repeat the entire procedure, or allow the cylinder contents to settle, pour out the overlying water when it is clear (making sure not to pour out any solids), and continue rinsing the sieve.
6. Allow the sediment to settle completely in the graduated cylinder and record the amount of sediment present. This measurement represents the *volume retained*. Also record any turbidity observed in the overlying water. The *volume retained* (in mL), subtracted from the original 50-mL aliquot, provides the volume that passed through the sieve, or *volume of fines* in 50 mL of sample. Multiplying this remainder by 2 gives the volume of fines in 100 mL, or *percent fines*. The formula can be stated as:

Percent Fines = (50 mL – Volume Retained in mL) X 2

pH

Sediment pH may be measured by two methods, depending on the type of pH probe that is used. When using either method, it is important to calibrate the pH meter prior to field use. The meter should be calibrated according to manufacturer's specifications with at least two buffers that will bracket the expected pH of the sediment samples. If the pH of a sediment sample falls outside the bracket of buffers in the initial calibration, the meter should be recalibrated with the proper buffers.

Sediment pH may be measured with a standard combination pH electrode by inserting the electrode directly into the sediment sample to a depth of approximately 2 cm. Record the measurement after the reading has stabilized. Standard combination pH electrodes are

sensitive and not very durable and care should be taken when inserting the electrode. An alternate method is described below.

A "soil" pH electrode contains a concentric ceramic junction above the reference contact. Sediment pH may be measured with this type of electrode as follows:

1. Collect approximately 5 g of sediment from the sample and place the aliquot in a small container such as a test tube.
2. Add approximately 5 mL of distilled water and mix completely.
3. Allow the mixture to settle for approximately 15 minutes.
4. Insert the electrode into the container so that the pH-sensitive bulb is immersed in the opaque sediment suspension and the reference contact remains in the relatively clear supernatant layer
5. Record the measurement after the reading has stabilized.

Rinse the electrode in distilled water after each use and store it in buffer between measurements.

REDOX POTENTIAL

Redox potential (or Eh) should be measured as soon as possible after sample collection due to the unstable nature of this parameter. Redox potential may be measured using a platinum electrode and combination pH/millivolt meter. The electrode is inserted directly into the sediment sample to a depth of approximately 2 cm. Record the measurement after the reading has stabilized.

The redox electrode should be calibrated prior to use with a solution of potassium ferrocyanide and potassium ferricyanide. Manufacturer's directions for preparation of the calibration solution are included with the electrode. This solution is poisonous and must be labeled, stored, and handled accordingly. Most electrodes should calibrate to a value near +192 millivolts using this calibration solution.

INTERSTITIAL SALINITY

The salinity of pore or interstitial water contained in a sediment sample may be measured directly in the field. An aliquot of the sediment sample is placed in a separate container not intended for chemical analysis and the sediment solids allowed to settle. The salinity of the overlying interstitial water may be measured directly using a salinometer. The salinometer should be calibrated prior to use according to manufacturers directions with a salinity standard of a concentration (in parts per thousand) close to that expected in the field. If the salinometer has a temperature compensation feature, the temperature of the interstitial water

should be measure prior to the salinity measurement and the salinometer adjusted accordingly.

Salinity of the interstitial water may also be measured indirectly from the measured conductivity and temperature of a sample. The conductivity meter should be calibrated prior to use with a known conductivity standard (e.g., in μS/cm) close to the conductivity expected at the sampling site and temperature measured prior to the conductivity measurement. Conductivity and temperature measurements may be used to calculate salinity according to methods outlined in Standard Method 2520B (APHA 1985).

REFERENCES

APHA. 1985. Standard methods for examination of waste and wastewater. 16th Edition. L.S. Clesceri, A.E. Greenberg, and R.R. Trussell (eds). American Public Health Association, Washington, DC.

STANDARD OPERATING PROCEDURE (SOP) SD-14

SEDIMENT CORING PROCEDURES USING A WILNER CORER

SCOPE AND APPLICATION

This SOP describes the procedures used to collect sediment samples with a Wilner corer. The project-specific field sampling plan (FSP) should stipulate the number of replicate samples (i.e., individual cores) or a total volume of sediment that needs to be collected at each station.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for sediment sampling includes the following:

- Wilner corer
- Core tubes and caps
- Sample collection tub
- Sample mixing bowl
- Ruler
- Plunger
- Scoop (for transferring sediment sample aliquots to the mixing bowl)
- Sample containers
- Soft-bristle nylon brush
- Hexane
- Deionized water
- Alconox
- Turkey baster.

PROCEDURES

Wear protective clothing as specified in the Investigation Area-specific health and safety plan when performing the sediment collection and sample processing.

Wilner Corer Deployment

1. Prior to deployment, clean the inside of the core tube with Alconox and a soft-bristle brush, followed by a hexane rinse, and then a deionized water rinse.
2. Clean the Wilner sampler with a soft-bristle brush and water from the Investigation Area.
3. Inspect the messenger and the auto-release mechanism to ensure good working order.
4. Attach the core tube to the Wilner sampler.
5. Slide the messenger up the rope away from the sampler to a distance that will not be submerged when the sampler is deployed.
6. Pull the plunger up to the auto-release mechanism and lock it in place.
7. Lower the sampler through the water column quickly, using the sampler's weight and momentum to penetrate the sediment.
8. If the water is too shallow to submerge the Wilner sampler, push the core tubes into the sediment to the prescribed depth by hand and plug the top end with a plastic cap to create a vacuum.

Wilner Corer Retrieval

1. After the sampler has penetrated the sediment, send down the messenger to release the plunger.
2. Once the plunger has released, retrieve the sampler.
3. Be prepared to place a cap on the bottom of the core tube before the grab sampler breaks the water surface. The cap may need to be placed on the bottom end of the tube while it is still under the surface of the water.
4. Lift the sampler above the surface of the water and rinse any sediment from the outside of the core tube and Wilner sampler using water from the Investigation Area.
5. Lift the sampler inboard and gently lower it into the sample collection tub.
6. Detach the core tube from the sampler.
7. Inspect the sample for acceptability. The following criteria must be satisfied or the sample will be rejected:

- The sampler is not filled with sediment above the top of the core tube.
- The sampler has penetrated the sediment to at least the depth specified in the project-specific FSP.
- There is no sign of sediment loss from the sediment depth that is required according to the project-specific FSP.

If a sample fails to meet the above criteria, discard the sediment sample in a manner that will not affect subsequent samples at that station or other possible sampling stations. Consecutive attempts should be as close to the original location as possible. Consecutive attempts on a river or stream should be located upstream of current.

8. To determine penetration depth, place a ruler against the side of the core tube and measure the distance from the top of the sediment to the lowest intact section of sediment at the bottom of the core tube.

Sample Removal and Processing

1. Use a turkey baster to siphon the overlying water off the top of the sediment sample in the core tube.
2. Use a plunger on the bottom of the core tube to push the contents of the sediment core upwards and extrude the sediment sample from the top of the tube.
3. Use a scoop or other utensil to collect sediment as it is extruded and place it in the sample mixing bowl. Handle the sediment sample only with clean, stainless steel utensils.
4. Ensure that only sediment from the desired depth is collected and included in the sample mixing bowl.
5. Discard any remaining sediment in the core tube away from the station.
6. If more than one sediment sample is required at a station, clean the core tube between samples, as described above.

STANDARD OPERATING PROCEDURE (SOP) SL-01

DECONTAMINATION OF SOIL SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either organic or inorganic materials. To prevent potential cross contamination of samples, all reusable soil sampling and processing equipment is decontaminated before each use. At the sample collection location, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All soil sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the OU4-specific health and safety plan (HASP).

Sampling equipment may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment (e.g., hand auger, split-spoon sampler) used for both analyte groups should follow the order of a detergent wash, rinse water, organic solvent rinses, and final rinse with water. Sample processing equipment (e.g., bowls, spoons) is rinsed with distilled/deionized water instead of with water from OU4.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following:

- Steam cleaner and collection basin (if required)
- 55-gal, Department of Transportation (DOT)-approved drums (if required)
- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or water from OU4 (i.e., potable water)
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial nonphosphate detergent
- Pesticide-grade ethanol and hexane (consult project-specific field sampling plan [FSP], as the solvents may vary by U.S. Environmental Protection Agency [EPA] region or state)
- 10 percent diluted nitric acid or hydrochloric acid (reagent grade) for inorganic contaminants (if required; see project-specific FSP)
- Baking soda (if required)
- Long handled, hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Personal protective equipment as specified in the HASP.

PROCEDURES

Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not one of the sample analytes. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is slightly more effective than other solvents, its use is discouraged because of its potential toxicity to sampling personnel. Always follow the procedures listed in the OU4-specific HASP when decontaminating sampling equipment (e.g., always stand upwind when using volatile solvents, wear appropriate gloves and safety glasses or goggles). Containerize all decontamination fluids for proper disposal, following procedures listed in the FSP.

The specific procedures for decontaminating soil sampling equipment and soil compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap water or water from OU4 to remove visible soil. This step should be performed on location for all equipment. After removing visible solids, set aside sampling equipment that does not need to be used again that day and see that it is thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap water or water from OU4. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles, using a back-and-forth motion. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double rinse the equipment with tap water or water from OU4 and set upright on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface. If acid and solvent rinses are not required by the FSP, skip to step 8.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle using a 10 percent acid solution. Double-rinse equipment with tap water or water from OU4 and set right-side-up on a stable surface to drain. If solvent rinses are not required by the FSP, skip to step 8.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). These solvents act primarily as a drying agent by scavenging water from the equipment surface and carrying it away, but they also work as a solvent for some organic contamination. Hand-augers must be held over the waste container and turned slowly so the stream of solvent contacts the entire surface. The sample apparatus may be turned on its side, and if applicable, opened to be washed more effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.
7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container, which may need to be equipped with a funnel. Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the

equipment was not thoroughly rinsed with ethanol or that the ethanol that was purchased was not free of water. When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the ethanol and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.

8. Do a final rinse with water from OU4 for the sampling equipment (i.e., hand-auger) and distilled/deionized water for the processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collection or processing equipment is precleaned at the field laboratory and transported to OU4, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.
10. After decontaminating all of the sampling equipment, dispose of the disposable gloves and used foil per the procedures listed in the project-specific FSP. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles for disposal at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda or containerized and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Metals and Conventional Parameters Only

The specific procedures for decontaminating soil sampling equipment and soil processing equipment are as follows:

1. Rinse the equipment thoroughly with tap water or water from OU4 to remove the visible soil. Perform this step on location for all equipment. Set aside any pieces that do not need to be used again that day see that they are thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap water or water from OU4. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.

3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double-rinse the equipment with tap water or water from OU4 (if an acid rinse is required) or with distilled/deionized water (if no acid rinse) and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle containing a 10 percent acid solution. Double-rinse equipment with distilled/deionized water and set right-side-up on a stable surface to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collecting or processing equipment is cleaned at the field laboratory and transported to OU4, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.
7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Drill Rig Sampling Equipment

1. Decontaminate sampling equipment before use, between samples and stations, and upon completion of sampling operations.
2. Equipment used during drilling operations should be decontaminated in the Exclusion Zone prior to transport to the Support Zone (refer to Investigation Area-specific HASP).
3. If the steam-cleaning location is in an area outside of the Exclusion Zone, remove loose soil on the drill rig, augers, drill pipe, and rods, and other large equipment at the drill location, then move the equipment directly to the steam-cleaning decontamination area for more thorough cleaning.
4. To decontaminate a drill rig, pressure wash with a steam cleaner using potable water rinse upon mobilization, between drilling locations, and upon demobilization. Cleaning water can generally be allowed to drain directly on the ground near the station (refer to the FSP).

5. To decontaminate auger, drill rods, and other down-hole tools, pressure wash with a steam cleaner and potable water rinse upon mobilization, between drilling locations, and upon demobilization. All decontamination fluids are to be containerized for proper disposal.
6. To decontaminate split-spoon and hand-auger samplers, follow the decontamination procedures listed above (the selected decontamination procedures is dependent upon analyte list provided in the project-specific FSP). To the extent possible, allow to air dry prior to sampling. If the split-spoon is not used immediately, wrap it in aluminum foil. All decontamination fluids are to be containerized for proper disposal.

STANDARD OPERATING PROCEDURE (SOP) SL-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SOILS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates (MS/MSDs), equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material) for soil samples. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if volatile organic compounds (VOCs) are to be analyzed. Definitions of all potential quality control samples are described below.

As part of the quality assurance and quality control (QA/QC) program, all field quality control samples will be sent to the laboratories blind. To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers that are required to complete the field quality control sample for the applicable analyte list must be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should only be recorded in the field logbook or field sampling forms. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent this from happening, select and mark regular samples on the chain-of-custody/sampling analysis request (COC) form or instruct the laboratory to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Prepare field quality control samples at least once per sampling event, and prepare certain types more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality

control sample per 20 is indicated and 28 samples are collected, prepare 2 quality control samples. The method of preparation and frequency of field quality control samples required for soil sampling activities are described below. These protocols must be followed, unless different frequency requirements are listed in the FSP and QAPP.

For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if VOCs are to be analyzed. The following table lists the possible quality control sample types and suggested frequencies for soil sampling programs (not all types of quality control samples will always be collected; see project-specific FSP and QAPP for actual quality control samples that need to be collected for a particular sampling event). A detailed explanation of each type of quality control sample with the required preparation follows.

Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling location	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling location	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling location	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples
Equipment rinsate blank	ER	Sampling location	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1:20 thereafter
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler
Environmental (transfer) blank	EB	Field	Bottle filled at sample location with deionized water	One per 20 samples
Standard reference material	SRM	Field laboratory or sampling location	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Collect field duplicate (or split) samples to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Prepare field duplicates by collecting two aliquots for the sample and submitting them for analysis as separate samples. Collect field duplicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of field duplicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Prepare field replicates by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Collect field replicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The project QA/QC coordinator will determine the actual number of field replicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The MS/MSD analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume that may be needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated soil stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of extra bottles collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Use equipment rinsate blanks to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Prepare equipment rinsate

blanks by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, then transferring the water to the appropriate sample containers and adding any necessary preservatives. Prepare equipment rinsate blanks for all inorganic, organic, and sometimes conventional analytes at least once per sampling event per the type of sampling equipment used. The project QA/QC coordinator will determine the actual number of equipment rinsate blanks prepared during an event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Submit bottle blanks along with soil samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, submit one bottle blank per lot of prepared bottles for analysis. If more than one type of bottle will be used in the sampling (e.g., HDPE or glass), then submit a bottle blank for each type of bottle and preservative. The project QA/QC coordinator will determine the actual number of bottle blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Use trip blanks to help identify whether contaminants may have been introduced during shipment of the soil samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two 40 mL VOC vials and tightly closing the lids. Invert each vial and tap lightly to determine if air bubbles exist. There should be no air bubbles in the VOC trip blank vials. If air bubbles are present, then note this information in the field logbook.

Transport the trip blanks unopened to and from the field in the cooler with the VOC samples. Label the trip blank and place it inside the cooler that contains newly collected VOC samples; it must remain in the cooler at all times. A trip blank must accompany samples at all times in the field. Send one trip blank (consisting of a pair of VOC vials) with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

The laboratory will use temperature blanks to verify the temperature of the samples upon receipt at the testing laboratory. The testing laboratory will prepare temperature blanks by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank must be included with each sample cooler shipped to the testing laboratory.

ENVIRONMENTAL BLANKS

Prepare the environmental (i.e., transfer) blank in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If you use unpreserved bottles, then you must add the appropriate preservative (e.g., for metals samples, use a 10 percent nitric acid solution to bring sample pH to 2 or less), if required. Collect environmental blanks at a minimum frequency of 1 in 20 samples. The project QA/QC coordinator will determine the actual number of environmental blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection location, fill the sample bottle with distilled/deionized water and then seal. Note the location from which the environmental blank was collected along with atmospheric conditions at the time of its collection in the field logbook. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

Reference materials (i.e., a standard reference material, a certified reference material, or other reference material are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. Reference materials have undergone multilaboratory analyses using a standard method which provides certified concentrations. When available for a specific analyte, Reference material samples provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several reference materials may be required to cover all analytical parameters. For all analytes where available, one reference material will be analyzed at a frequency of one per 50 samples. The project QA/QC coordinator will determine the actual number of reference materials analyzed during a project on a case-by-case basis (consult the project-specific FSP

and QAPP, as the requirements on frequency of reference material analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SL-04

FIELD CLASSIFICATION OF SOIL

SCOPE AND APPLICATION

This SOP establishes the minimum information that must be recorded in the field to adequately document surface soil sampling and soil borehole advancement activities performed during field exploration. The surface soil sampling or borehole log form must be filled out completely for each station.

This SOP presents the field classification of soils to be used by Integral field staff. In general, Integral has adopted the procedures provided in American Society for Testing and Materials (ASTM) Method D-2488-00, Standard Practice for Description and Identification of Soils (attached). ASTM D-2488-00 uses the Unified Soil Classification (USC) system for naming soils. Field personnel are encouraged to study these procedures prior to initiation of fieldwork.

Soil descriptions should be precise and comprehensive without being verbose. The overall impression of the soil should not be distorted by excessive emphasis on minor constituents. In general, the similarities of consecutive soil samples should be emphasized and minor differences de-emphasized. These descriptions will be used to interpret potential contaminant transport properties, rather than interpret the exact mineralogy or tectonic environment. We are primarily interested in engineering and geochemical properties of the soil.

Soil descriptions should be provided on the surface soil field collection form or in the soil description column of the Integral's soil boring log for each sample collected. If there is no difference between consecutive soil samples, subsequent descriptions can be noted as "same as above" or minor changes such as "increasing sand" or "becomes dark brown" can be added.

The format and order of soil descriptions should be as follows:

- Group symbol (in the Unified Symbol column)
- USC name (should be identical to the ASTM D-2488-00 Group Name with the appropriate modifiers)
- Minor components
- Color
- Moisture
- Additional descriptions.

EQUIPMENT AND REAGENTS REQUIRED

- Surface soil field collection form or borehole log form (see SOP SL-06, *Logging of Soil Boreholes*)
- Munsell® soil color chart.

PROCEDURES

The USC is an engineering properties system that uses grain size to classify soils. The first major distinction is between fine-grained soils (more than 50 percent passing the No. 200 sieve [75 μm/0.0029 in.]) and coarse-grained soils (more than 50 percent retained by the No. 200 sieve). Small No. 200 sieves are necessary to classify soils near the cutoff size.

1. Fine-grained soils are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-00. If these tests are used, include the results in the soil description. If these materials are encountered, perform at least one complete round of field tests for the subject property, preferably at the beginning of the field characterization. The modifiers "fat" and "lean" are used by ASTM to describe soils of high and low plasticity. The soil group symbols (e.g., CL, MH) already indicate plasticity characteristics, and these modifiers are not necessary in the description. Soils with high plasticity can be emphasized by describing them as "silty CLAY with high plasticity." Plasticity, for example, is an important descriptor because it is often used to interpret whether an ML soil is acting as either a leaky or a competent aquitard. For example, an ML soil can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.
2. Coarse-grained soils are classified as either predominantly gravel or sand, with the No. 4 sieve (4.75 mm/0.19 in.) being the division. Use modifiers to describe the relative amounts of fine-grained soil, as noted below:

Description	Percent Fines	Group Symbol
Gravel (sand)	<5 percent	GW, GP (SW, SP)
Gravel (sand) with silt (clay)	5–15 percent	Hyphenated names
Silty (clayey) gravel (sand)	>15 percent	GM, GC (SM, SC)

The gradation of a coarse-grained soil is included in the specific soil name (e.g., fine to medium SAND with silt). Estimating the percent of size ranges following the group name is encouraged for mixtures of silt sand and gravel. Use of the modifiers "poorly graded" or "well graded" is not necessary, as they are indicated by the group symbol.

Show a borderline classification with a slash (e.g., GM/SM). Use this symbol when the soil cannot be distinctly placed in either soil group. Also use a borderline symbol when describing interbedded soils of two or more soil group names when the thickness of the beds are approximately equal, such as "interbedded lenses and layers of fine sand and silt." Do not use a borderline symbol indiscriminately. Make every effort to place the soil into a single group. (One very helpful addition to the soil log form description is the percentage of silt/sand/gravel. Even if the geologist did not have sufficient time to properly define the soil, this percentage breakdown allows classification at a later date).

3. Precede minor components, such as cobbles, roots, and construction debris with the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). Use the word "occasional" to describe random particles of a larger size than the general soil matrix (i.e., occasional cobbles, occasional brick fragments). The term "with" indicates definite characteristics regarding the percentage of secondary particle size in the soil name. It is not to be used to describe minor components. If a nonsoil component exceeds 50 percent of an interval, state it in place of the group name.
4. Give the basic color of a soil, such as brown, gray, or red. Modify the color term with adjectives such as light, dark, or mottled, as appropriate. Especially note staining or mottling. This information, for example, may be useful to establish water table fluctuations or contamination in boreholes. The Munsell® soil color chart designation is the Integral color standard. These charts are readily available and offer a high degree of consistency in descriptions between geologists.
5. Define the degree of moisture present in the soil as dry, moist, or wet. Moisture content can be estimated from the criteria listed in Table 3 of ASTM D-2488-00.
6. If observed, note such features as discontinuities, inclusions, joints, fissures, slickensides, bedding, laminations, root holes, and major mineralogical components. Note anything unusual. Additional soil descriptions may be made at the discretion of the project manager or as the field conditions warrant. The surface soil field collection and soil boring log forms list some optional descriptions, as does Table 13 of the ASTM standard. The reader is referred to the ASTM standard for procedures of these descriptions.

The contact between two soil types must be clearly marked on the surface soil field collection or soil boring log forms. If the contact is obvious and sharp, draw it in with a straight line. If

it is gradational, use a slanted line over the interval. In the case where it is unclear, use a dashed line over the most likely interval.

For drilling activities, the field geologist, who has the advantage of watching the drilling rate and cuttings removal and can talk with the driller in real time, has a much better chance of interpreting the interval than someone in the office.

ATTACHMENT 1. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-µm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved Feb. 10, 2000. Published May 2000. Originally published as D 2488 – 66 T. Last previous edition D 2488 – 93$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols.*

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula*.

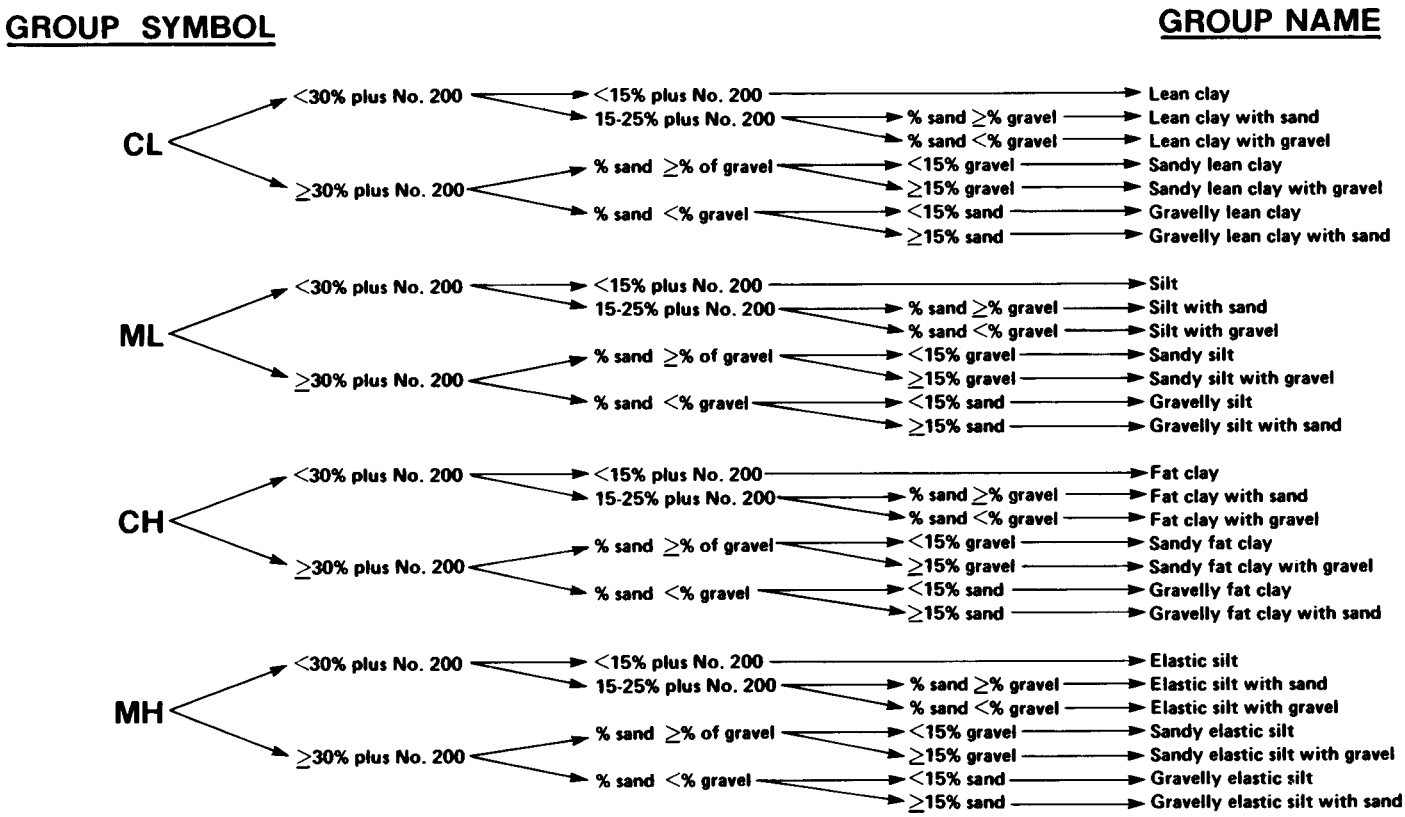
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens*.

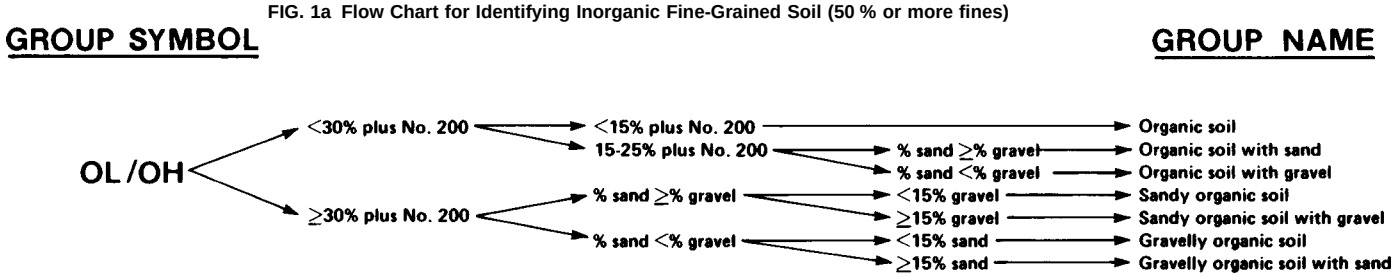
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

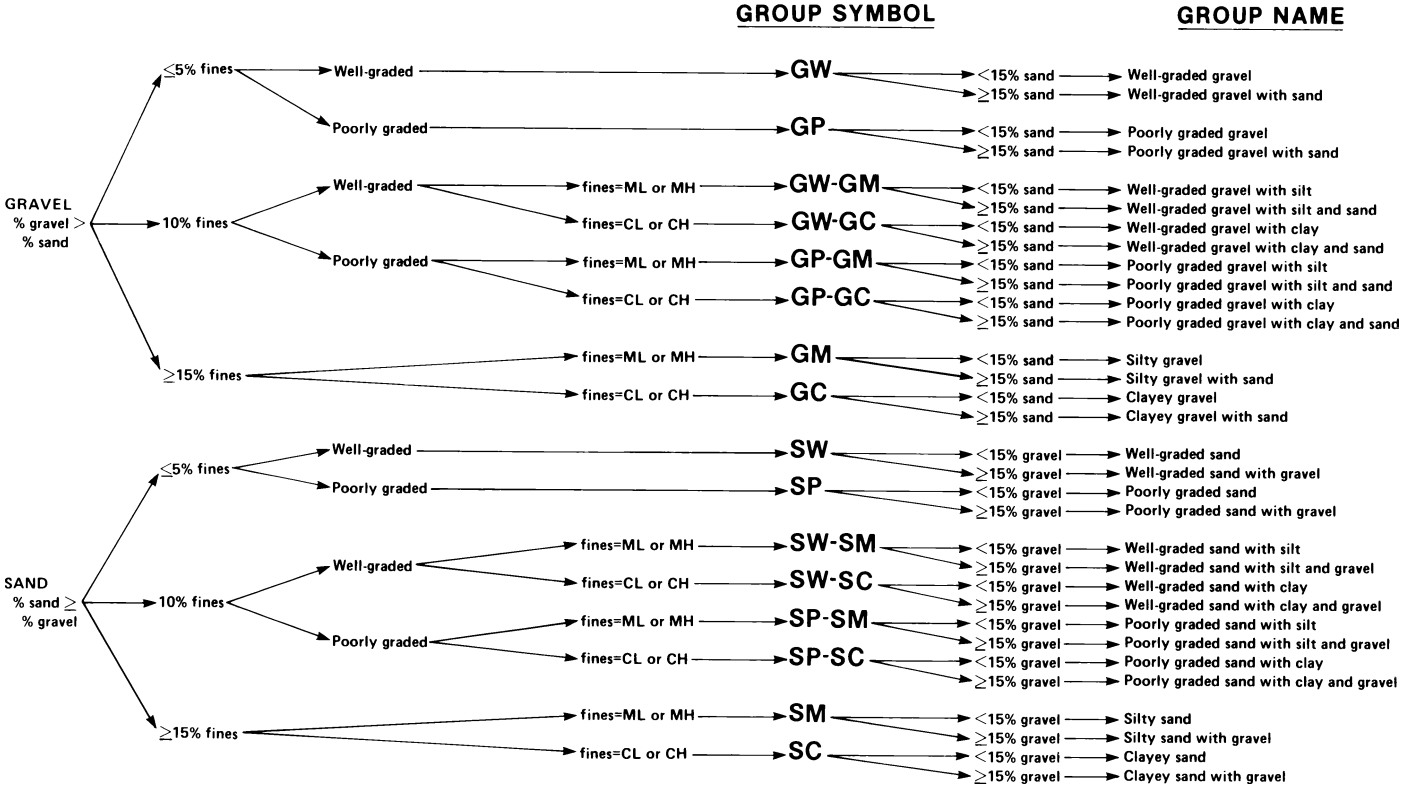
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

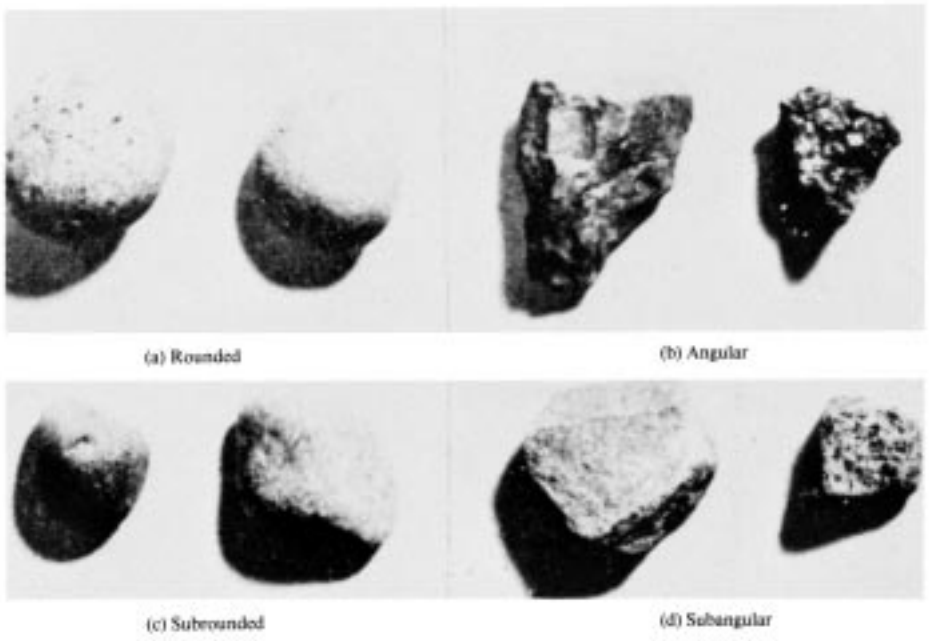


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

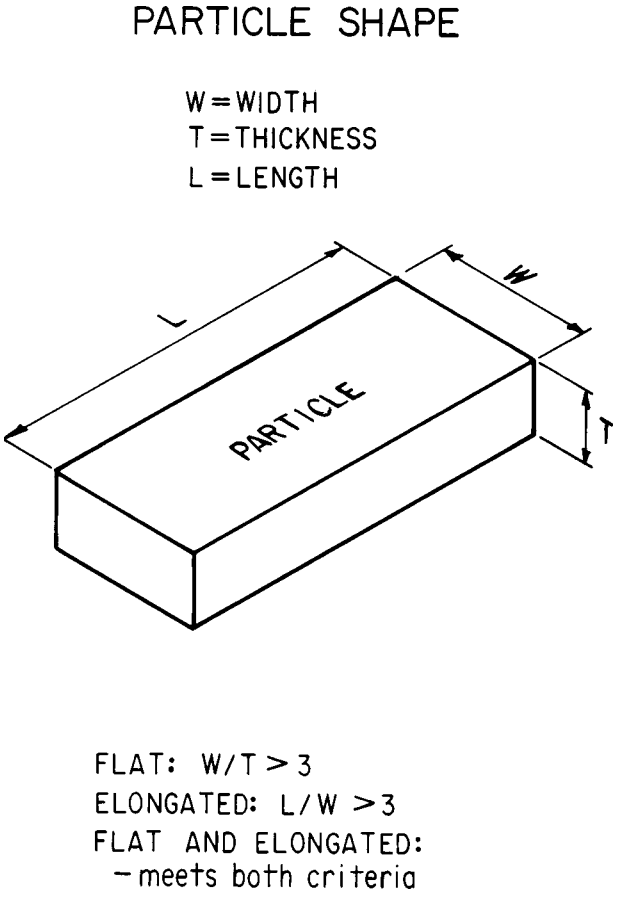


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as “Sandy Lean Clay (CL)”; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; “Poorly Graded Sand with Silt (SP-SM)”; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken

shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993$^{\epsilon 1}$) that may impact the use of this standard.

(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

STANDARD OPERATING PROCEDURE (SOP) SL-05

SURFACE SOIL SAMPLING

SCOPE AND APPLICATION

This SOP defines and standardizes the collection of surface soil samples (e.g., 0 to 2 in. below ground surface). Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

The procedures listed below may be modified in the field upon the agreement of the lead sampler and field personnel, based on field conditions, after appropriate annotations have been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral's SOP on methanol preservation of soil samples. Record all pertinent information on Integral's surface soil sampling field data form or field logbook.

EQUIPMENT AND SUPPLIES REQUIRED

- Decontaminated sampling tool (stainless-steel shovel, scoop, trowel, or spoon)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Ziploc® bags
- Camera
- Tape measure
- Field logbook, surface soil field collection form, and pens
- Project-specific field sampling plan (FSP) and health and safety plan (HASP)
- Personal protective equipment (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HASP)
- Decontamination equipment.

PROCEDURES

1. Locate the sample station as directed in the project-specific FSP. Label containers with sample tags prior to filling in accordance with Integral's SOP on sample labeling (SOP-AP04). If analytical testing will be performed for volatile organic compounds (VOCs), collect the VOC sample first (with a minimum of disturbance) by placing the sample into the container with a minimum amount of headspace and sealed tightly.
2. Don a new pair of nitrile gloves and expose the soil surface by clearing an approximately 1 ft^2 area at the sampling location of any rocks, other solid material/debris, or organic material greater than approximately 3 in. in size. Note any material removed from the sampling location in the field logbook.
3. Using a decontaminated stainless-steel sampling tool, excavate soil to the depth specified in the work plan.
4. If required for analysis, first collect VOC samples (prior to any homogenization) from a discrete location, placing the samples in the appropriate containers. Label sample containers before filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
5. Place additional sample material in a decontaminated plastic or stainless-steel mixing bowl.
6. Describe the soil in accordance with ASTM D2488-00 (see Integral's SOP on field classification of soils, SOP SL-04).
7. Thoroughly mix and homogenize the sample using disposable equipment or a decontaminated stainless-steel spoon until the color and texture are consistent throughout.
8. If required for analysis, first collect samples for grain-size tests before any large rocks are removed from the homogenized soil.
9. Identify any rocks or other solid material/debris that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized soil volume, note it on the surface soil field collection form or in the field logbook, and then discard.
10. Remove samples of the homogenized soil from the mixing bowl with the decontaminated stainless steel spoon and place in the appropriate size sample container. Do not touch the sample with your gloves. Fill the sample container with soil to just below the container lip, and seal the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling.
11. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate.

12. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
13. Photograph sample location and document it in the logbook.
14. Decontaminate all sampling equipment according to Integral's SOP on decontaminating equipment for soil sampling (SOP SL-01) and in accordance with the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SL-06

LOGGING OF SOIL BOREHOLES

SCOPE AND APPLICATION

This SOP describes how to complete a Soil Boring Log form, which must be completed for Integral projects where soil boring techniques are performed during field exploration. A correctly completed form contains all of the information that must be recorded in the field to adequately characterize soil boreholes.

These procedures are adapted from ASTM D-2488-00. Field staff are encouraged to examine ASTM D-2488-00 in its entirety. This SOP represents minor modifications to emphasize environmental characterizations rather than geotechnical characterizations, for which the standards were written. Because each environmental project is unique and because job requirements can vary widely, the minimum standards presented may need to be supplemented with additional technical descriptions or field test results. However, all soil boring field logs, regardless of special project circumstances, must include information addressed in this SOP to achieve the minimum acceptable standards required by Integral.

LOG FORM INFORMATION

Project Number—Use the standard contract number.

Client—Identify the name of the client and the project location.

Location—If stations, coordinates, mileposts, or similar markers are applicable, use them to identify the location of the project. If this information is not available, identify the facility (e.g., 20 ft NE of Retort #1).

Drilling Method—Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, cable tool) and the name of the drill rig (e.g., Mobil B 61, CME 55).

Diameter—Provide the diameter of the borehole. If the borehole has variable diameters, provide the depth interval for each diameter.

Sampling Method—Identify the type of sampler(s) used (e.g., standard split spoon, Dames & Moore sampler, grab).

Drilling Contractor—Provide the name of the drilling contractor.

Integral Staff—Enter the name(s) of Integral staff members performing logging and sampling activities.

Water Level Information—Provide the date, time, depth to static water, and casing depth. Generally, water levels should be taken each day before resuming drilling and at the completion of drilling. If water is not encountered in the boring, this information should be recorded.

Boring Number—Provide the boring number. A numbering system should be developed prior to drilling that does not conflict with other Study Area information, such as previous drilling or other sampling activities.

Sheet—Number the sheets consecutively for each boring and continue the consecutive depth numbering.

Drilling Start and Finish—Provide the drilling start and finish dates and times.

For consecutive sheets, provide (at a minimum) the job number, boring number, and sheet number.

TECHNICAL DATA

Sampler Type—Provide the sampler type (e.g., SS = split spoon, G = grab).

Depth of Casing—Enter the depth of the casing below ground surface immediately prior to sampling.

Driven/Recovery—Provide the length that the sampler was driven and the length of sample recovered in the sampler. This column would not apply to grab samples.

Sample Number/Sample Depth—Provide the sample number. The sample numbering scheme should be established prior to drilling. One method is to use the boring number and consecutive alphabetical letters. For instance, the first sample obtained from boring MW-4 would be identified as 4A, the second would be identified as 4B, and so on. Another method for sample identification is naming the boring number with the depth. For example, the sample from Boring 1 at 10 ft would be labeled B1-10'. The depth of the sample is the depth of the casing plus the length to the middle of the recovered sample to the nearest 0.1 ft. Typically, split spoon samplers are 18 in. long. Samples should be obtained from the middle of the recovered sample. The depth of the sample with the casing at 10 ft would then be 10.7 ft.

Number of Blows—For standard split-spoon samplers, record the number of blows for each 6 in. of sampler penetration. A typical blow count of 6, 12, and 14 is recorded as 6/12/14. Refusal is a penetration of less than 6 in. with a blow count of 50. A partial penetration of 50 blows for 4 in. is recorded as 50/4". Total blows will be recorded for nonstandard split spoons (e.g., 5-ft tube used for continuous sampling).

Blank Columns—Two blank columns are provided. Use these columns for OU4-specific information, usually related to the chemicals of concern. Examples for a hydrocarbon location would be sheen and photoionization detector readings of the samples.

Depth—Use a depth scale that is appropriate for the complexity of the subsurface conditions. The boxes located to the right of the scale should be used to graphically indicate sample locations as shown in the example.

Surface Conditions—Describe the surface conditions (e.g., paved, 4-in. concrete slab, grass, natural vegetation and surface soil, oil-stained gravel).

Soil Description—Enter the soil classification and definition of soil contacts using the format described in SOP SL-04, *Field Classification of Soil*.

Comments—Include all pertinent observations. Drilling observations might include drilling chatter, rod-bounce (boulder), sudden differences in drilling speed, damaged samplers, and malfunctioning equipment. Information provided by the driller should be attributed to the driller. Information on possible contaminants might include odor, staining, color, and presence or absence of some indicator of contamination. Describe what it is that indicates contamination (e.g., fuel-like odor, oily sheen in drill cuttings, yellow water in drill cuttings).

ATTACHMENT 1. SOIL BORING LOG FORM

integral consulting inc.
319 SW Washington St., Suite 1150
Portland, OR 97204
(503) 284-5545

STATION NUMBER
PROJECT
LOCATION
PROJECT NUMBER
LOGGED BY

SAMPLE INFORMATION						STRATA	DESCRIPTION
Sample ID	Depth	Time	Tag No.	% Recov.	Depth (Feet)		USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, weathering, cementation, geologic interpretation, etc.
					2--		
					4--		
					6--		
					8--		
					10--		
					12--		
					14--		

DRILLING CONTRACTOR
DRILLING METHOD
SAMPLING EQUIPMENT
DRILLING STARTED
COORDINATES
SURFACE ELEVATION
DATUM

Location Sketch

ATTACHMENT 2. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-µm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved Feb. 10, 2000. Published May 2000. Originally published as D 2488 – 66 T. Last previous edition D 2488 – 93$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols.*

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula*.

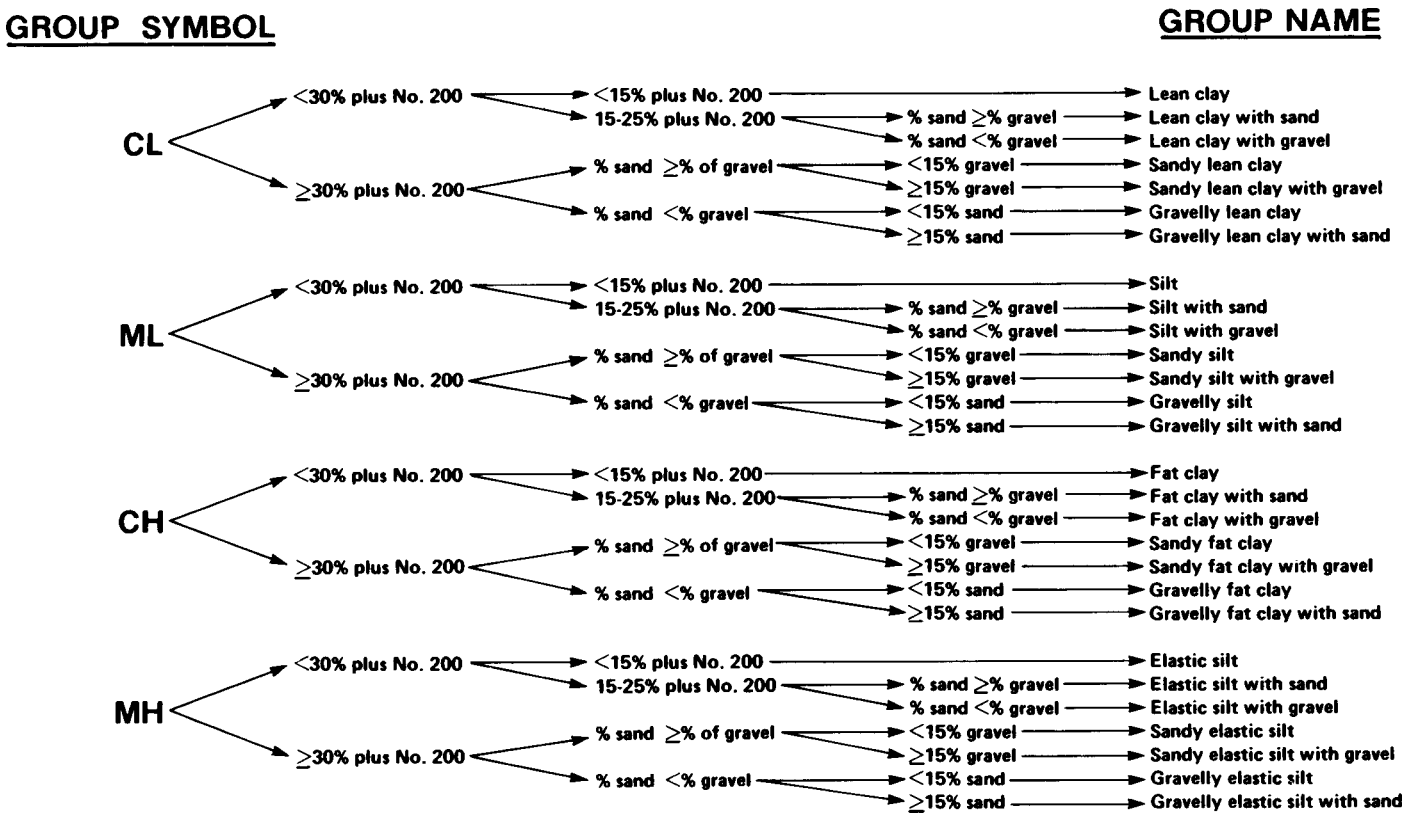
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens*.

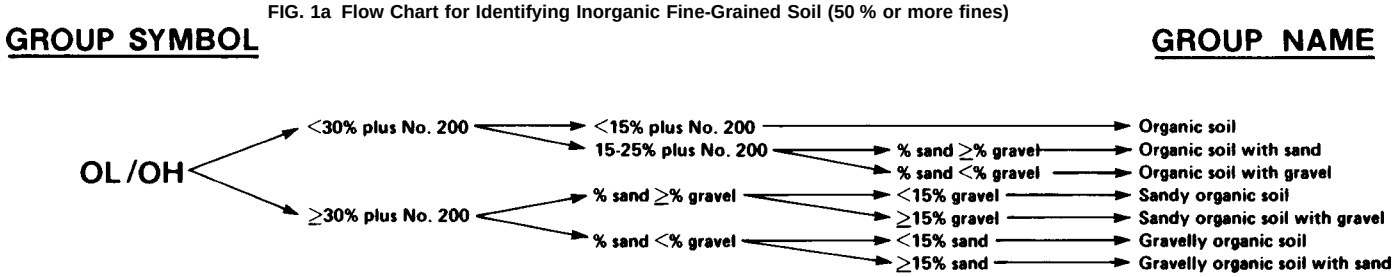
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

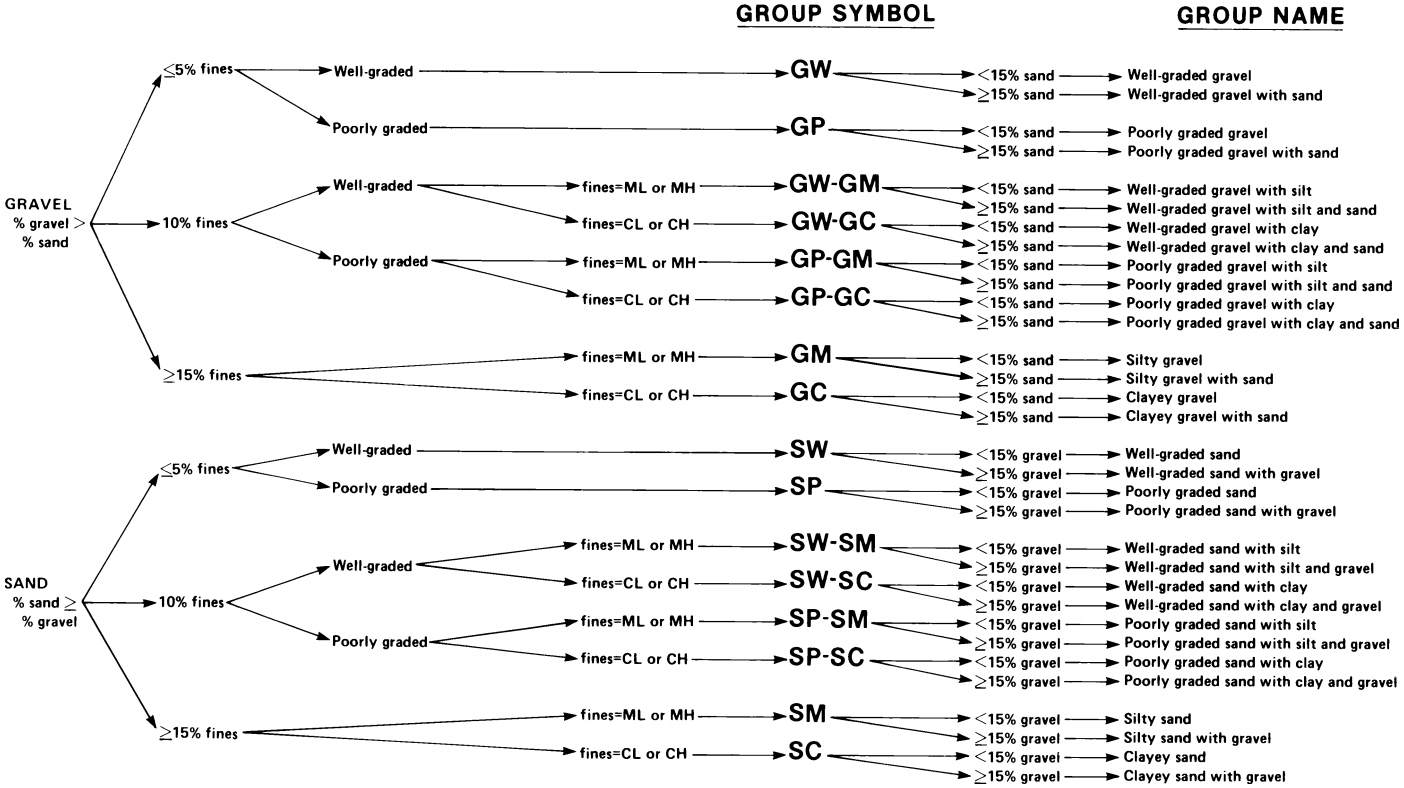
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

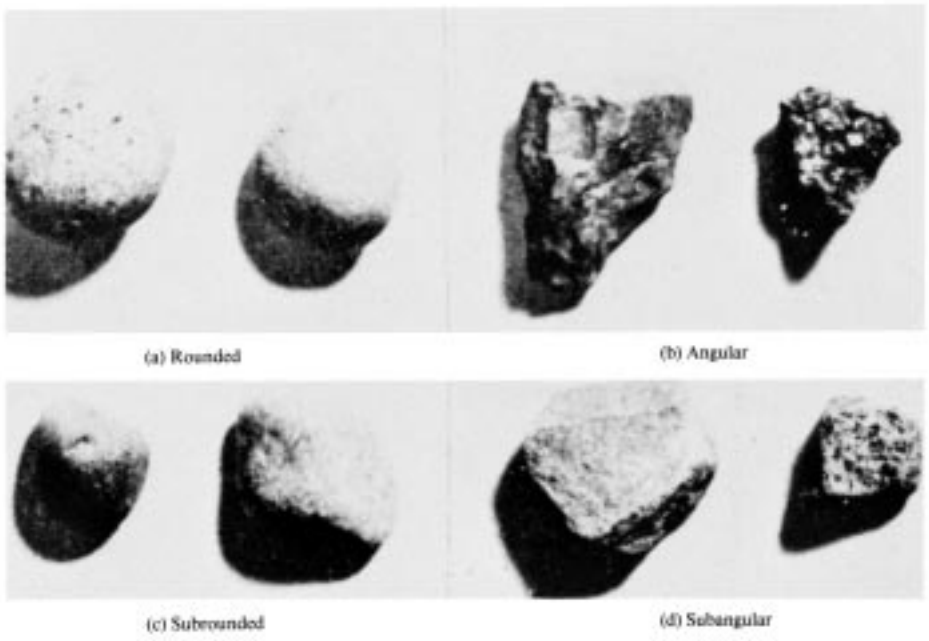


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

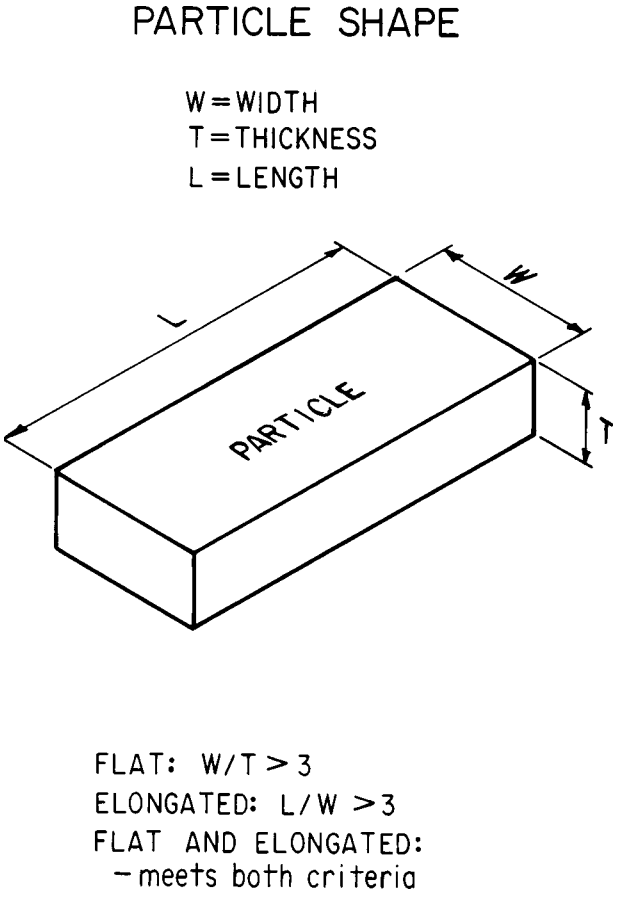


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as “Sandy Lean Clay (CL)”; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; “Poorly Graded Sand with Silt (SP-SM)”; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken

shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993$^{\epsilon 1}$) that may impact the use of this standard.

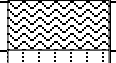
(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

ATTACHMENT 3. FIELD CLASSIFICATION OF SOILS, BASED ON UNIFIED SOIL CLASSIFICATION SYSTEM AND ASTM STANDARD D-2488

integral consulting inc.

Field Classification of Soils, Based on Unified Soil Classification System and ASTM Standard D-2488

Major Divisions		Symbol	Pattern	General Soil Description
Coarse-Grained Soils (More than 1/2 of soil >No. 200 sieve size)	**Gravels**	GW		Well-graded gravels or gravel-sand mixtures, little to no fines
		GP		Poorly-graded gravels or gravel-sand mixtures, little to no fines
		GM		Silty gravels or gravel-sand-silt mixtures
		GC		Clayey gravels or gravel-sand-clay mixtures
	Sands	SW		Well-graded sands or gravel-sand mixtures, little to no fines
		SP		Poorly-graded sands or gravelly sands, little to no fines
		SM		Silty sands, sand-silt mixtures
		SC		Clayey sands, sand-clay mixtures
Fine-Grained Soils (More than 1/2 of soil <No. 200 sieve size)	**Silts**	ML		Inorganic silts with slight plasticity
		MH		Inorganic elastic silts
		OL		Organic elastic silts
	Clays	CL		Inorganic clays of low to medium plasticity, lean clays
		CH		Inorganic clays of high plasticity, fat clays
		OH		Organic clays of medium to high plasticity
Highly Organic Soils		Pt		Peat, sample composed primarily of vegetable tissue

Soil Classification Notes

Groundwater, First Observed

Groundwater, Static

Sampling Equipment

SS	Split Spoon
ST	Shelby Tube
GS	Geoprobe® Macrocore Sampler

Sheen Types

NS	No Sheen
LS	Light Sheen
MS	Moderate Sheen
HS	Heavy Sheen

Sample Moisture

Dry	No moisture, dry to touch
Moist	Damp, but no free water
Wet	Visible free water

Sample Plasticity (Fine-Grained Soils)

Non-Plastic -	Cannot be rolled at any moisture content.
Low -	Can barely be rolled, lump cannot be formed when drier than plastic limit.
Medium -	Can easily be rolled, lump crumbles when drier than plastic limit.
High -	Can easily be rolled, but takes considerable time to reach the plastic limit. Lump can be formed without crumbling when drier than the plastic limit.

Particle Size Range (Coarse-Grained Soils)

Gravel - Fine, Coarse

Sand - Fine, Medium, Coarse

STANDARD OPERATING PROCEDURE (SOP) SL-07

SUBSURFACE SOIL SAMPLING

SCOPE AND APPLICATION

The following procedures are designed to be used to collect subsurface soil samples using a hand auger or direct-push drill rig. *All underground utilities must be located and cleared prior to drilling or excavating.* Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

Based on field conditions, the procedures listed below may be modified in the field upon agreement of the field team leader and project management, after appropriate annotations have been made in the project-specific field logbook. If specialized sampling methods (e.g., Encore®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral SOP SL-08 on methanol preservation of soil samples. Record all pertinent information in the Integral field logbook, subsurface soil field collection form, or boring log (as appropriate).

EQUIPMENT AND SUPPLIES REQUIRED

- Subsurface sampling equipment (e.g., hand auger, direct-push drill rig [e.g., Geoprobe®], stainless-steel spade) (consult project-specific field sampling plan [FSP] for kind of equipment to be used for a specific field event)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Resealable plastic bags (e.g., Ziploc®)
- Camera
- Tape measure
- Logging table
- 6-mil visqueen and duct tape for covering the logging table
- Aluminum foil

- 55-gallon drums for decontamination waters and excess soil (separate drums for liquid and solid wastes) if required by the project-specific FSP
- Field logbook, subsurface soil field collection form, and/or soil boring form, and pens
- Project-specific FSP and health and safety plan (HSP)
- Personal protective equipment (PPE) (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Photoionization detector (PID), if required by the project-specific FSP or HSP
- Global positioning system (GPS), if required by the project-specific FSP
- Decontamination equipment.

HAND AUGER SAMPLER

The following procedures are designed to be used during the general operation of a hand auger sampler. The procedures listed below may be modified in the field upon agreement of the field team leader and drill operators, based on field conditions, after appropriate annotations have been made in the field logbook.

1. Locate the sample station as directed in the project-specific FSP. Place sample labels on the sample container prior to filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
2. Place plastic sheeting adjacent to the sampling location.
3. Advance the hand auger into subsurface soil.
4. Empty soil from the first interval (as specified in the project-specific FSP) from the hand auger into a decontaminated stainless steel bowl and cover the bowl with aluminum foil. Continue advancing the hand auger until the next appropriate sample interval has been completed.
5. Screen the soil sample for volatile organic compounds (VOCs) using a PID if required by the project-specific FSP.
6. Photograph each interval with depth and location markers visible in the photograph, if applicable.
7. Log the soils in accordance with SOP SL-04 (*Field Classification of Soils*).
8. If VOC samples are required (see project-specific FSP), collect them prior to homogenizing (i.e., mixing) the sample. Collect the VOC sample (with a minimum of disturbance) by placing the sample into the container with no headspace and sealing it tightly. If an Encore® sampling device is specified in the project-specific FSP, follow the sample collection guidelines provided by the manufacturer.

9. (a) If the soil sample is to be a discrete sample (see project-specific FSP), collect soil from the hand auger using a decontaminated stainless-steel spoon and place the sample into a decontaminated stainless-steel bowl. Homogenize the soil to a consistent color and texture.

   (b) If additional sample volume is required to perform the analyses specified in the project-specific FSP, place multiple soil samples collected from nearby locations (it is important to keep the distance between multiple soil borings as close as possible; the maximum distance will be specified in the project-specific FSP) from the same depth interval into a composite sample in a single decontaminated stainless-steel bowl. When a sufficient volume of soil has been obtained, homogenize all of the soil in the bowl to a consistent color and texture using a decontaminated spoon.

10. Discard rocks or other solid material/debris, found in the homogenized soil that are greater than 0.5 in. in diameter after positively identifying them, determining their percentage contribution to the homogenized soil volume, and noting it in the field notebook.

11. Remove samples of the homogenized soil from the compositing bowl and place in the appropriate size sample container. Fill the sample container with soil to just below the container lip, and seal the container tightly.

12. Decontaminate all sampling equipment in accordance with SOP SL-01 and the project-specific FSP.

13. Repeat the process described above for all subsequent sample intervals.

14. Complete the appropriate field books, field data sheets, and quality assurance and quality control (QA/QC) documentation. Record any deviations from the specified sampling procedures or any obstacles encountered.

15. Backfill the borehole with remaining hand auger soil cuttings or place the cuttings in a properly labeled 55-gallon drum, as specified in the project-specific FSP. If soil cuttings are placed in a 55-gallon drum, backfill the borehole with bentonite hole plug pellets and hydrate the pellets with potable water.

16. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate. Collect GPS coordinates of the sample location if specified in the project-specific FSP.

DIRECT-PUSH DRILL RIG

The following procedures are designed to be used during the general operation of direct-push drill rig (e.g., Geoprobe®). The procedures listed below may be modified in the field upon agreement of the field team leader and drill operators, based on field conditions, after

appropriate annotations have been made in the field logbook. The direct-push drill rig will be operated by a licensed drilling contractor.

The direct-push drilling technique hydraulically pushes tools into the ground to collect soil samples. Direct-push drilling techniques can be used to collect soil samples to depths of 30–100 ft, depending on drilling conditions. In addition to soil sample collection, direct-push techniques can be used to collect soil gas samples, reconnoiter groundwater samples, and install small-diameter monitoring wells.

Soil samples can be collected using two types of Macrocore® samplers, open tip and closed tip. These samplers are typically either 4 ft long by 1.5 in. inside diameter (i.d.) or 5 ft long by 2.5 in. i.d. These samplers have a tubular design and utilize acetate liners to collect the soil samples. The following sections of this SOP describe how to collect soil samples using open-tip and closed-tip Macrocore® samplers.

Open-Tip Sampler

The open-tip sampler is typically used in soils that are cohesive (e.g., stiff silts and clays), where the soil boring is stable and stays open when the sampler and rods are removed from the ground.

1. Ensure all underground utilities are cleared prior to initiating drilling activities.
2. Position the direct-push drill rig over the sample station and remove any surface material that will interfere with sampling. Note in the field logbook any surface material that is removed prior to sampling.
3. Determine the interval to be sampled and install a new clean liner into the open tip Macrocore® sampler.
4. Push the sampler to the bottom of the appropriate sample interval.
5. Retract the rods and Macrocore® sampler.
6. After the Macrocore® sampler has been brought to the surface, remove the liner from the sampler, cap both ends of the liner, and inspect it.
7. After the soil sample is judged to be acceptable, label the sample liner with the station identifier, depth interval, and soil orientation (i.e., arrow pointing toward uppermost soil interval).
8. Place the capped sample liner on a new piece of aluminum foil on the logging table and split the liner open with a hook or utility knife. Process the sample in accordance with the "General Sampling Procedures" listed below.
9. Repeat Steps 2–8 for each subsequent sample interval.

Closed-Tip Sampler

The closed-tip sampler is typically used to collect soil samples that are noncohesive (e.g., sandy materials), where the soil boring is unstable and collapses when the rods and sampler are removed from the ground.

1. Ensure all underground utilities are cleared prior to initiating drilling activities.
2. Position the direct-push drill rig over the sample station and remove any surface material that will interfere with sampling. Note in the field logbook any surface material removed prior to sampling.
3. Determine the interval to be sampled and install a drive point and a new clean liner into the closed-tip Macrocore® sampler.
4. Push the rods and sampler to the top of the appropriate sample interval.
5. Retract the rods to release the drive point.
6. Push the sampler to the bottom of the appropriate sample interval.
7. Retract the rods and Macrocore® sampler.
8. Once the soil sample has been brought to the surface, remove the liner from the sampler, cap both ends of the liner, and inspect it.
9. After the soil sample is judged to be acceptable, label the sample liner with the station identifier, depth interval, and soil orientation (i.e., arrow pointing toward uppermost soil interval).
10. Place the capped sample liner on a new piece of aluminum foil on the logging table and split the liner open with a hook or utility knife. Process the sample in accordance with the "General Sampling Procedures" listed below.
11. Repeat Steps 2–10 for each additional sample interval.

General Sampling Procedures

1. After the liner has been split open, screen the soil sample for VOCs using a PID if required by the project-specific FSP.
2. Log the soils in accordance with SOP SL-04 (*Field Classification of Soils*).
3. Photograph each section of the soil boring with appropriate orientation, depth, and location markers visible in the photograph, if specified in the project-specific FSP.

4. If VOC samples are required (see project-specific FSP), collect them prior to sample removal from the liner. Collect the VOC sample (with a minimum of disturbance) by placing the sample into the container with no headspace and seal it tightly. If an Encore® sampling device is specified in the project-specific FSP, follow the sample collection guidelines provided by the manufacturer.
5. Remove the soil from the liner using a decontaminated stainless-steel spoon and place the soil in a decontaminated compositing bowl and thoroughly mix and homogenize the sample using a decontaminated spoon until the color and texture are consistent throughout.
6. (a) If the soil sample is to be a discrete sample (see project-specific FSP), collect soil from the liner using a decontaminated stainless-steel spoon and place the sample into a decontaminated stainless-steel bowl. Homogenize the soil to a consistent color and texture.

   (b) If additional sample volume is required to perform the analyses specified in the project-specific FSP, place multiple soil samples collected from nearby locations (it is important to keep the distance between multiple soil borings as close as possible; the maximum distance will be specified in the project-specific FSP) from the same depth interval into a composite sample in a single decontaminated stainless-steel bowl. When a sufficient volume of soil has been obtained, homogenize all of the soil in the bowl to a consistent color and texture using a decontaminated spoon.
7. Discard rocks or other solid material/debris found in the homogenized soil that are greater than 0.5 in. in diameter after positively identifying them, determining their percentage contribution to the homogenized soil volume, and noting it in the field notebook.
8. Remove samples of the homogenized soil from the compositing bowl and place in the appropriate size sample container. Fill the sample container with soil to just below the container lip, and seal the container tightly.
9. Repeat the process described above for subsequent sample intervals.
10. Complete the appropriate field books, field data sheets, and QA/QC documentation. Record any deviations from the specified sampling procedures or any obstacles encountered.
11. Backfill the borehole with remaining direct-push sampler cuttings or place the cuttings in a properly labeled 55-gallon drum, as specified in the project-specific FSP. If soil cuttings are placed in a 55-gallon drum, backfill the borehole with bentonite grout (mixed to the manufacturer's specifications) or bentonite hole plug pellets and hydrate the pellets with potable water.

12. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate. Collect GPS coordinates of the sample location if specified in the project-specific FSP.
13. Decontaminate all sampling equipment in accordance with SOP SL-01 and the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SW-01

DECONTAMINATION OF SURFACE WATER SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP defines and standardizes Integral's methods for decontamination of field sampling equipment for collecting surface water samples to ensure sample integrity and minimize contamination during sample handling.

This SOP utilizes and augments the procedures outlined in the San Francisco Estuary Institute's *Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001), *Interagency Field Manual for the Collection of Water-Quality Data* (USGS various dates), and U.S. Environmental Protection Agency (EPA) Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996). Clean sampling techniques designed for trace metals will be used for the collection of filtered and unfiltered water samples.

Samples may be analyzed for organic compounds, metals, nutrients, and conventional analytes for the surface water sampling events, according to the project-specific sampling and analysis plan (SAP).

To prevent cross-contamination of samples, all reusable surface water sampling equipment will be decontaminated before each use. Decontamination of field sampling equipment can be done in the field or in a commercial laboratory. Depending on the project's complexity and analytical reporting limits (see project-specific SAP), sampling equipment may need to be decontaminated at a qualified laboratory. It is strongly discouraged to decontaminate sampling equipment in the field due to the high risk of contamination. Thorough decontamination procedures should be followed under controlled conditions at the laboratory. However, it is necessary to perform certain decontamination steps in the field.

Set up a decontamination station on the Investigation Area in a clean location upwind of sampling locations, or perform decontamination in the field office, under a laboratory hood if available. Store decontaminated equipment away from contaminated areas and in a manner that will prevent recontamination prior to use.

When handling decontamination chemicals, follow all relevant procedures outlined in the Investigation Area-specific health and safety plan.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following, depending on the target analyte and sampling equipment:

- Plastic brushes with rigid bristles
- Properly labeled squirt bottles
- 5-gal plastic bucket
- Tap water
- Alconox®, Liquinox® detergent, or equivalent
- Pesticide-grade decontamination solvents (e.g., ethanol and methanol, according to the project-specific SAP, as the solvents may vary by EPA region or state)
- Nitric acid (5 percent)
- Hydrochloric acid (10 percent) if nutrients are being analyzed
- Deionized water (analyte-free; received from testing laboratory)
- Sealable waste container equipped with a funnel
- 1 gal sealable plastic bags
- 2.5 L amber glass bottles.

DECONTAMINATION PROCEDURES

Decontamination methods vary depending on whether the samples collected will be analyzed for conventional analytes, organic chemicals, or trace metals.

Conventional Analytes and First Use

The following procedure is used when sampling for conventional analytes such as chloride, sulfate, sodium, and calcium. It is also used for new equipment and for equipment that is being used for the first time at a Investigation Area. Conventional analytes have the simplest decontamination procedure because they tend to be highly soluble in water and detergent solutions, and do not tend to sorb significantly to the surface of the sampling equipment.

For collection of lake water samples at different depths from the same location, equipment needs to be rinsed only with water from the Investigation Area three times between stations following an initial decontamination. Similarly, for collection of samples from rivers where stations are close to one another spatially and temporally, only a rinse with water from the Investigation Area is necessary. The steps are as follows:

1. Rinse the equipment thoroughly with tap water.

2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (some detergents contain surfactants that are analytes) and set aside to drain.
4. Rinse the equipment three times with water from the Investigation Area immediately prior to collecting the sample.

Organic Chemicals

The following procedure is used for decontaminating equipment (e.g., Kemmerer sampler) used to collect surface water that will be analyzed for organic chemicals. Two organic solvents are used in the procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). The second organic solvent is hydrophobic (e.g., methanol) and is intended to dissolve any organic chemicals on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific SAP). The choice of solvents also depends on the material the equipment is made from (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol and hexane are sometimes slightly more effective than other solvents, their use is discouraged because of toxicity to sampling personnel. The decontamination procedure is as follows:

1. Rinse the equipment thoroughly with tap water or water from the Investigation Area.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap water or water from the Investigation Area. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap water or water from the Investigation Area and set aside to drain.
4. Rinse the equipment with ethanol dispensed from a squirt bottle and let the excess solvent drain into a waste container equipped with a funnel (ethanol acts primarily as a drying agent, but also works as a solvent for some organic contamination). Rinse the inside of the sampling equipment that comes in contact with sample water. Set the equipment in a clean location and allow it to air dry. In cold temperatures, it may take a long time for equipment to dry. In this case, it is important to remove all water from the surface by thoroughly rinsing with a more volatile solvent such as acetone. In hotter temperatures, use a less volatile water solvent (e.g., isopropanol).

5. Rinse the air-dried equipment with methanol dispensed from a squirt bottle and let the excess solvent drain into the waste container. Methanol acts as the primary solvent, but it is insoluble with water. If water beading occurs, it means that the equipment was not thoroughly rinsed with ethanol or the equipment was not given sufficient time to dry completely. Rinse the inside of the sampling equipment that comes in contact with water from the Investigation Area. In hotter climates, use a less-volatile solvent such as methanol. When the equipment has been rinsed thoroughly, set it in a clean location and allow the solvent to evaporate before storing or using it.
6. Close the solvent waste container when not in use and store it in a secure place.
7. Transfer the waste to empty solvent bottles and dispose of it at a licensed facility.

Trace Metals

In addition to the following decontamination procedures, personnel collecting water samples must be aware of other sources of contamination. Sources commonly encountered in the field include lead batteries used to power pumps, metal objects such as tools, and gasoline cans. To the extent possible, these items should be removed from the sample collection area and the sampling equipment, and anyone collecting the samples should avoid handling these items beforehand. Wear vinyl clean-room gloves (e.g., Oak class 100, powder free) when handling sampling equipment that will be used to collect surface water samples for trace metals analysis. Discard gloves between stations or if they come into contact with any materials known or likely to be contaminated.

The following procedures should be used for decontaminating equipment used to collect surface water samples for trace metals (e.g., Teflon™ tubing, Teflon™ churn splitter, connectors and adapters made of Teflon™ or other similar material, or plastic stands used for holding sample tubing). This procedure is not intended for containers in which samples will be stored and/or shipped to the laboratory for analysis.

1. Rinse the equipment thoroughly with tap water or water from the Investigation Area.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap water or water from the Investigation Area. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution. Fill bottles about halfway with detergent solutions and shake for a few minutes. Pump the detergent solution through any tubing for a few minutes. Small parts can be placed in large-mouth jars that have tight lids and shaken with the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (detergents will neutralize the nitric acid) and set it aside to drain.

4. Clean all equipment surfaces that come into contact with water samples using a 5 percent nitric acid solution for at least 30 minutes. Place small items, such as Teflon™ water intakes, in plastic containers filled with 5 percent nitric acid. Fill sampling containers/bottles with 5 percent nitric acid solution and allow to stand. Cover the containers and keep them away from potential contamination sources.
5. Either pump acid solution through tubing, or leave it static in the tubing for the same duration.
6. Drain all equipment thoroughly and flush with at least three volumes of laboratory deionized water (not deionized water from the grocery store).
7. Drain thoroughly and flush with at least three volumes of water from the Investigation Area before collecting a sample.

PROCEDURES USED TO DECONTAMINATE SAMPLING DIAPHRAGM PUMPS

The following procedure is used for samples to be analyzed for trace metals and conventional analytes. Two types of pumps are commonly used for collecting water samples, peristaltic and diaphragm. For peristaltic pumps, only the tubing needs to be cleaned according to the above procedure. It is best to keep precleaned short lengths of tubing for each station when using the peristaltic pump. For diaphragm pumps, the procedure is as follows:

1. Using two short pieces of tube on the pump, place both ends in a 1-gal container with detergent solution and circulate the solution through the system for 2 minutes.
2. Purge the system with about 1 gal of laboratory deionized water, keeping the outflow tubing over a waste bucket. Do not recirculate this solution. Repeat the 1 gal deionized water purge.
3. Connect the two ends of the short tubes with a decontaminated plastic coupler and keep it sealed until sampling time.
4. When ready to sample, remove the short tubing protecting the inlet of the pump, connect the tubing used for sampling to the pump, and purge the system with water from the Investigation Area for 2 minutes, or with enough water to rinse the entire system (i.e., pump head and tubing) immediately before collecting the sample.

REFERENCES

David, N., D. Bell, and J. Gold. 2001. Field sampling manual for the regional monitoring program for trace substances. San Francisco Estuarine Institute, San Francisco, CA.

USEPA. 1996. Method 1669 – Sampling ambient water for trace metals at EPA water quality criteria levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC.

USGS. [various dates]. National field manual for the collection of water-quality data: U.S. Geological Survey techniques of water-resources investigations, Book 9, Chaps. A1-A9. http://pubs.water.usgs.gov/twri9A. Accessed February 5, 2008. http://water.usgs.gov/owq/FieldManual/index.html#Citation. U.S. Geological Survey.

STANDARD OPERATING PROCEDURE (SOP) SW-20

STORMWATER GRAB SAMPLING

SCOPE AND APPLICATION

This SOP defines and standardizes the methods for collecting water from storm drains, outfalls, flumes, or at sheet flow locations. Outfalls are locations where stormwater exits a facility property, including through pipes, ditches, swales, and other structures that transport stormwater. Sheet flow is dispersed runoff that flows offsite (e.g., through a grassy area or across a parking lot).

The goal of this SOP is to ensure that the highest quality, most representative data are collected, and that these data are comparable to data collected by other programs that follow guidelines of the U.S. Environmental Protection Agency (EPA). The procedures detailed in this SOP were adapted from EPA's Industrial Stormwater Monitoring and Sampling Guide (USEPA 2009) and Ecology (2010). The procedures provided in this SOP may be modified by the field team leader if warranted by site conditions (e.g., configuration of outfall).

Not all of the sampling methodologies discussed in this SOP may be required for a given project. The specific sample collection techniques and associated sampling equipment will be detailed in the project-specific sampling and analysis plan (SAP). This SOP describes the methods for collecting either grab or composite stormwater samples.

STATION ACCESS

Prior to entering areas at which an outfall is located (such as private beaches or embayments, or near private docks), it may be necessary to acquire property access permission from the landowner. Be sure to secure such permission, including any written agreements, in advance of the sampling program.

If the outfall is covered and the cover needs to be removed to obtain the sample, then contact site or regulatory personnel to provide approvals to open the outfall cover. Integral personnel are not trained in confined space entry and will not, under any circumstances, enter a manhole, outfall, or stormwater vault. Provisions should be made to hire a confined space entry-licensed subcontractor if sampling equipment needs to be installed at confined locations.

SUMMARY OF METHOD

To collect stormwater samples for standard chemical and conventional analyses, two kinds of sampling approaches can be used depending upon the project's needs: direct sample collection into the laboratory-supplied bottle or sample collection using a pole with a decontaminated sampling container at the end to collect the sample. This SOP is intended for the collection of discrete grab samples. If composite samples are required by the project-specific SAP follow procedures provided in SOP SW-21.

SUPPLIES AND EQUIPMENT

- Field filtering equipment: peristaltic pump, filters and dedicated disposable tubing (if dissolved constituents are required, consult the project-specific SAP for types of filters, and sample volume requirements)
- Extension pole with attachment clips or strapping tape and auxiliary sample collection container (if necessary)
- Water quality meter (consult project-specific SAP for list of required water quality parameters) and calibration solutions (if necessary)
- Reed manhole tool or crowbar (if necessary to open outfall cover)
- Stakes and flagging tape for marking access to remote or overgrown outfalls
- Machete, weed whacker, or branch loppers for cutting vegetation
- Ziploc® bags
- Sample tags/labels and appropriate documentation (e.g., chain-of-custody forms, custody seals)
- Laboratory-supplied sample containers with preservatives (see project-specific SAP), insulated coolers, and ice
- Camera
- Handheld differential global positioning unit
- Field logbook, stormwater sample collection form (Attachment 1), and indelible pens or markers
- Project-specific SAP, and project-specific health and safety plan (HSP)
- Project-specific personal protective equipment (e.g., foul-weather gear, safety glasses, footwear appropriate for the conditions at the sampling locations[steel-toed boots, non-slip boots], powder-free nitrile gloves, and any other items required by the site HSP)

- Safety equipment including a first aid kit and flashlight in case of darkness under storm conditions.
- Cell phone, two way radio, or satellite phone if working in remote areas
- Decontamination material (typically Alconox® and distilled water)
- Coolers with ice.

PROCEDURES

The sampling team should comprise at least two people to conduct the sampling, handle the filtration (if necessary; see project-specific SAP), collect the water quality parameters, and keep track of sample logging and processing. When collecting any type of stormwater sample, it is imperative that the sample be collected before the stormwater reaches the receiving water. Therefore, the sampling team should be on standby and closely monitor the local weather forecast.

Equipment Preparation

Reusable equipment (extension pole/collection vessel apparatus) must be decontaminated prior to use and between sample locations. The decontamination procedure is as follows:

1. Rinse equipment with site water
2. Wash with a soap (e.g., Alconox®) solution
3. Rinse with distilled water (commercial potable water)
4. Final rinse with laboratory-grade deionized water.

Use dedicated tubing and dedicated laboratory-supplied sample bottles for field filtration to avoid performing decontamination procedures between stations.

Sample Handling

Hands must be gloved when collecting and handling samples, as described above. Field staff will wear appropriate non-contaminating, disposable, powderless nitrile gloves during the entire sampling operation. This includes any and all sample handling that may occur during sample packing and shipping (see SOP AP-01).Gloves will be changed frequently, usually with each change in task (wearing multiple layers of gloves allows rapid glove changes).

Gloved hands are required for all operations that involve equipment that comes into contact with the sample, including the following activities:

- Handling the sample bottle
- Handling the filter (if needed)
- Collection of water quality parameters.

All samples are to be stored in coolers with ice at approximately 4°C. The sampling team leader is responsible for maintaining sample integrity throughout the sampling event.

Field Observations

All field observations are to be recorded in field logbook.

1. Record the sample number and outfall/discharge identifier.
2. Record the date and duration of the storm event sampled and collect station location coordinates
3. Record weather conditions at the time of sampling and last known rain fall event(s). Document the duration between of the storm event sampled and the previous storm event. Note whether the discharge resulted from rainfall or snowmelt or general operations at the site such as parking lot wash.
4. Obtain rainfall measurement or estimate (in inches). Consult the local area weather monitoring program (e.g., airports).
5. Record the location and sketch the basic drainage areas on the map for the outfall, and if applicable, note whether concentrated flows comingle with sheet flow (see Sheet Flow Sample Collection Section for examples). Photo-document the outfall.
6. Visually assess the stormwater (i.e., color, odor, clarity, floating solids, settled solids, suspended solids, foam, oil sheen) and note observations in the field logbook.
7. Note if there is stormwater entering the outfall or sheet flows from neighboring properties and commingling with the stormwater being collected.
8. If possible, determine where the storm drain is sending the runoff (e.g., to a municipal storm water system, to a combined sewer system, to a separate sanitary sewer system, or directly to a nearby water body).

Sample Collection

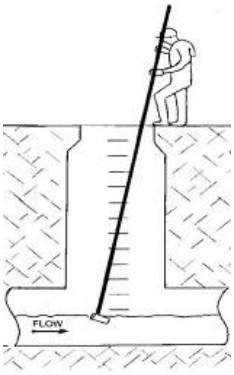
Stormwater Grab Sample Collection from an Outfall or Pipe

For runoff flowing through a pipe into a ditch or receiving water, the sample should be collected directly from the pipe using the following procedures:

1. Wear disposable powder-free nitrile gloves; never touch the inside of the bottle or lid. Change gloves between each sample.

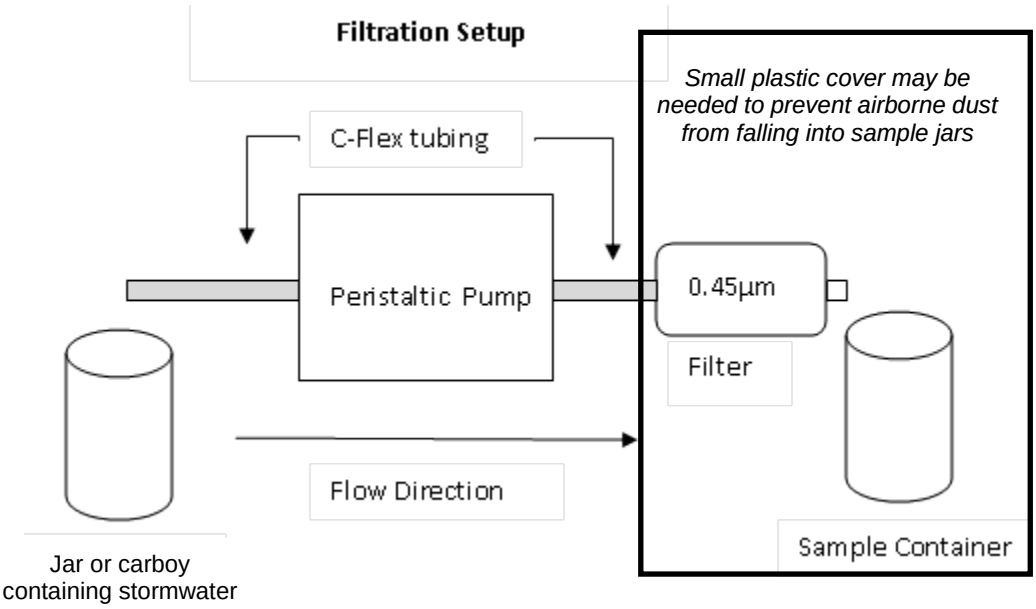
2. Obtain a new laboratory-supplied sample container to collect a grab sample from the outfall. Label sample containers before filling in accordance with the project-specific SAP.
3. Collect stormwater sample directly from the pipe.

4. In areas where samples cannot be easily collected directly from the outfall, a decontaminated 500 mL polypropylene sample collection bottle attached to an extension pole can be lowered and/or extended to the desired sampling location.



5. For both scenarios, hold the sample bottle or extension pole with collection bottle below the outfall in the flow of the water with the mouth pointing upstream and below the water surface.
6. Sample from a turbulent section of the central part of the flow. Be careful not to touch the bottle to the sides of the outfall or the bottom substrate.
7. Fill the sample bottle nearly to the top (meniscus almost at the rim) by holding the opening into the flow of water; do not rinse or overfill the bottles.
8. Transfer contents to the appropriate laboratory supplied containers, if necessary.

9. If volatile organic compound (VOC) analysis is required (see project-specific SAP), collect samples for VOCs in 40-mL volatile organic analyte (VOA) vials making absolutely certain that there are no bubbles adhering to the sides or top of the VOA container and that there is no headspace in the container. Be sure to check that the condition of samples is acceptable in the VOA containers before leaving each sampling site. If any air bubbles are present, collect a new VOA sample using a fresh sample container (i.e., VOA vials come with a known volume of preservative).
10. If oil and grease analysis is required (see project-specific SAP), fill the glass sample bottle directly from the discharge; never collect this sample in a container first and then transfer to the sample bottle because oily residue will collect along the inside of the first collection bottle and make the sample results inaccurate.
11. Repeat steps 2 through 10 until all sample containers are filled.
12. If filtered metals and dissolved organic carbon analysis is required (see project-specific SAP), place the filter (see project-specific SAP for pore size of the filter) in-line near the tubing outlet to filter samples immediately before the water is discharged into the sample bottle. Follow procedures below:
    - Collect sufficient stormwater from sample location in a clean laboratory supplied sample container or carboy (e.g., 10 L to 20 L glass carboy). That is, several liters of water may need to be composited to collect enough material to rinse filter and fill sample containers.
    - Set up peristaltic pump with C-flex tubing and new filter (or appropriate type and pore size filter). The filter is installed in-line at the sample tubing outlet to filter samples immediately before the water is discharged into the sample bottle (see figure below).
    - If a pre-cleaned filter cartridge containing laboratory deionized water is used, drain the storage solution inside the filter, and flush the entire sample tubing and filter assembly with sample water. Discard this first "rinse" of sample water. After rinsing the filter and sample tubing, fill the sample bottle to the "neck" with filtered sample water. If there is a risk of sample jars freezing during storage, allow enough head space in the sample jar to accommodate expansion of ice and therefore prevent potential loss of samples.

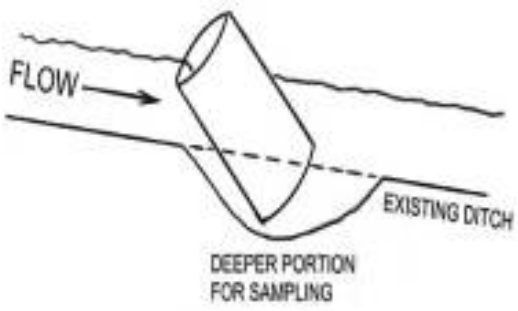


13. If required by the project-specific SAP, collect an aliquot of water for measuring water quality parameters (e.g., temperature, dissolved oxygen, pH, turbidity, and specific conductivity) in a clean container (e.g., bucket) before filtering. Measure water quality parameters *immediately after* the sample is collected.
14. If the area is dusty, use a small plastic cover to protect the filtered water outflow into sample jars (see diagram above).
15. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
16. Package and ship samples according to SOP AP-01.

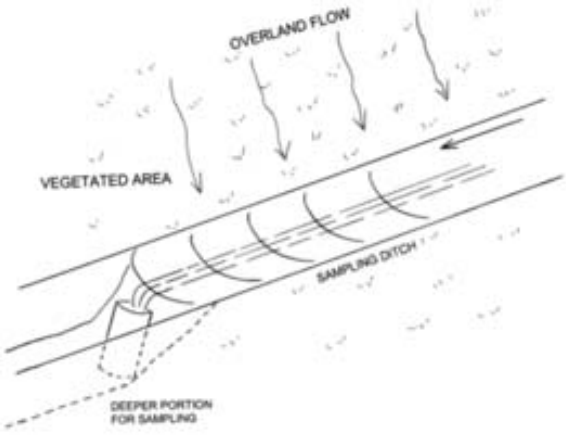
Sheet Flow Sample Collection

In some areas of the site, it may be difficult to obtain a sample because the runoff drains as sheet flow before it becomes concentrated enough for sampling. If the flow is too shallow to directly fill a collection bottle, concentrate the sheet flow in one of the following three ways (as described in USEPA 2009):

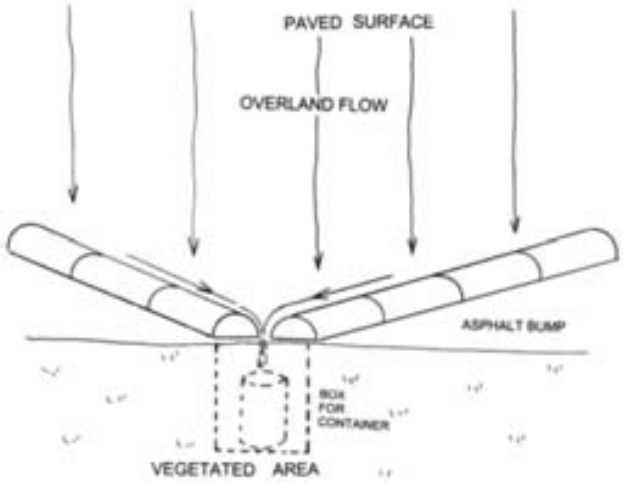
- Excavate a small depression in an existing ditch (take care to not introduce sediment into your sample). If feasible, line the ditch with concrete of plastic to prevent cross contamination.



- Install a trough, gutter or ditch to intercept and concentrate flow.



- Install "speed" bumps to convey and concentrate a large area of flow.



Make modifications for sheet flow sampling during dry conditions in preparation for sampling. Exercise care in choosing appropriate materials that will not be a source of contaminants of interests. Sample collection procedures will follow the same methods as described above for outfall and pipe sampling.

Water Quality Measurements

If specified in the project-specific SAP, measurements of physical and chemical water parameters may need to be collected at stormwater stations. Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field if feasible). In addition, measurements of temperature and turbidity can be collected accurately only in the field.

1. Use a YSI 650/6600 multi-probe (or equivalent meter) for measuring surface water parameters, such as temperature, pH, dissolved oxygen, conductivity, oxidation-reduction potential, and turbidity (consult project-specific SAP for list of required water quality parameters). If rental equipment is used, the unit will arrive pre-calibrated. Check it daily for proper functioning and drift. The proper handling for the multi-probe is described in detail in SOP SW-06.
2. Place the water quality meter probe directly into the surface water body at the station location at the desired water depth. If direct immersion is not possible, which is often the case, use a clean plastic bucket to collect samples for water quality analyses (e.g., pH, temperature, and conductivity). Rinse the bucket twice with the water from the station prior to measuring water quality parameters.
3. Record the measurement(s), the name(s) of the person(s) taking the measurement, and the field equipment used to make that measurement the field logbook and on any field forms used during the sampling event. Keep equipment maintenance and calibration records in logbooks and field records so that the procedures are traceable.

REFERENCES

Ecology. 2010. How to do stormwater sampling; a guide for industrial facilities. Publication #02-10-071. Washington State Department of Ecology, Water Quality Program, Olympia, WA. http://www.ecy.wa.gov/pubs/0210071.pdf

USEPA. 2009. Industrial stormwater monitoring and sampling guide. Final Draft. EPA 832-B-09-003. U.S. Environmental Protection Agency, Office of Water, Wastewater Management Program, Washington, DC. http://www.epa.gov/npdes/pubs/msgp_monitoring_guide.pdf

STORMWATER SAMPLE COLLECTION FORM

Station ID _____ Project Name: _____

Sample ID _____ Project Number:_____

Date _____ Field Staff:_____

Time _____

Field Parameters

pH	Temperature (C)	Conductivity (uS/cm)	Turbidity (NTU)	Comments
_____	_____	_____	_____	_____

Sampling Device:

Filter:

Sample Containers **Collection Time**_____

	Type		Preservative	Analytical Method	QA Remarks
	☐Amber	☐Poly (1 L)			
	☐Amber	☐Poly (250 mL)			
	☐Amber	☐Poly (250 mL)			
	☐Amber	☐Poly (250 mL)			
	☐Amber (1 L)	☐Poly			
	☐Amber (1L)	☐Poly			

General condition of outfall / Comments:

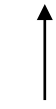
Flow Conditions:

Weather Conditions:

Last known Rainfall Event:

Photo Number	Direction	Description

Outfall Sketch



Samplers' Signature_____ Date _____

integral engineering pc

APPENDIX E

AERIAL PHOTOGRAPHS


08-05-1938


05-11-1942

05-18-1944


04-16-1952


07-11-1955


05-04-1960


09-07-1962


10-08-1964


03-30-1968


05-07-1973


05-10-1982


04-14-1986


09-22-1991


04-14-1999


04-24-2002


03-21-2012

APPENDIX F

BIOLOGICAL SURVEY APPROACH

Appendix F

Biological Survey Approach for Delineation of the Mean High Water Level for Study Area OU4

This appendix presents an approach for a biological survey to be performed to confirm or refine the mean high water level (MHWL) presented in the draft *Characterization Work Plan: Study Area Operable Unit 4 Flood Control Areas, NYSDEC Project ID 851046, Corning New York* (OU4 Work Plan) prepared by Integral Engineering, P.C. (Integral). This biological survey provides a process to demarcate soils and sediment areas within OU4.

Regulatory Background

The Technical Guidance for Site Investigation and Remediation (DER-10; NYSDEC 2010) defines sediment as:

> …unconsolidated particulate material found at the bottom of lakes, rivers, streams and other water bodies at bed elevations equal to or lower than the mean high water level as defined in 6 NYCRR 608.1(r). [Note: Materials present in enclosed sumps, sewers or piping systems not accessible to fish and wildlife and not forming any benthic or aquatic habitat are not considered sediments for the purpose of comparison to DEC's *Technical Guidance for Screening Contaminated Sediment*.]

New York State Regulation 6 CRR-NY 608.1(r) states that the MHWL "distinguishes between predominantly aquatic and predominantly terrestrial habitat." The regulation also states that this demarcation is to be determined primarily based on available hydrologic data, and secondarily based on vegetative characteristics.

DEC's Technical Guidance for Screening and Assessment of Contaminated Sediment (NYSDEC 2014) is:

> …applicable to sediments that comprise the substrate of waterbodies up to the mean high water line. In regards to wetlands, these guidelines can be applied to sediments in permanently inundated wetlands such as marshes and swamps that border waterbodies. They may not be applicable to wetlands that are only occasionally submerged, or are more soil-like in composition, however, that applicability should be determined on a case-by-case basis.

Integral used the available hydrologic data and additional relevant information to establish a best estimate of MHWL, as discussed in the OU4 Work Plan. The biological survey will be performed as a second step to confirm/refine the estimated demarcation between terrestrial and aquatic habitat, and to assess the applicability of the sediment screening guidance in areas that are only occasionally submerged or periodically inundated.

Approach and Technical Basis

A biological survey will be performed to evaluate terrestrial and aquatic vegetation (consistent with 6 NYCRR 608.1(r)) to confirm or refine the MHWL presented in the OU4 Work Plan. The evaluation of vegetative cover will be used to distinguish conditions most representative of OU4 soil present in terrestrial habitat, versus soil and sediment present in aquatic habitat.

To characterize soil and sediment areas, the biological survey approach is designed to identify habitat conditions that may have the potential of sustaining aquatic habitat. While areas containing water during periodic inundation may support certain benthic species for short durations, sustained maintenance of established and diverse benthic communities requires the consistent presence of aquatic habitat (i.e., regular presence of water). Many benthic organisms are sessile (e.g., bivalves) or largely limited in mobility (e.g., larval arthropods, gastropods, aquatic oligochaetes). In order to survive and reproduce on a long-term basis (e.g., year after year), benthic species in these communities require the continual presence of water. The biological survey approach therefore considers the presence of aquatic vegetation requiring the continual presence of water as a surrogate for aquatic habitat conditions required for maintaining benthic communities.

Methods

The biological survey entails the use of established scientific methods for performing vegetation surveys for plant communities (e.g., USGS 1995; Causton 1988; Kent and Coker 1992; Zonneveld 1988) and the vegetation classification methods described in NYSDEC's Freshwater Wetlands Delineation Manual (Manual; NYSDEC 1995). While use of the vegetation classification methods from the Manual is proposed, this biological survey is not intended as a wetlands delineation. As described in Section 5.4.2 of the OU4 Work Plan, a separate field effort will be conducted to complete a wetlands delineation in OU4.

- The survey will be performed when the flow in the Chemung River (monitored at the Corning U.S. Geological Survey [USGS] gaging station number 01529950, which is situated approximately 0.25 mile upstream of OU4) is at or below 845 cubic feet per second, the mean monthly summertime (July, August, September) flow, based

on 39 years of monitoring[1], such that water level is not prohibitive of completion of the survey).

- Qualified field biologists representing Corning Incorporated and NYSDEC will perform the biological survey together, achieving consensus of survey results in the field.
- The approach starts by mapping of the estimated MHWL for the Chemung River and Post Creek in OU4 as presented in the OU4 Work Plan. Global positioning system (GPS) coordinates will be obtained and used as waypoints for this boundary and will be uploaded in handheld GPS units with submeter accuracy for use in the field.
- Using the handheld units containing the GPS coordinates, a qualified field biologist will use stick flags to mark Integral's currently estimated MHWL across OU4.
- Every 300 ft, starting at the western end of OU4, the field biologist will use stakes and flagging to delineate a 3-m-wide by 10-m-long rectangle centered on the MHWL, such that 5 m extends upslope (toward the flood control berm) and 5 m extends downslope (toward the Chemung River or Post Creek). At a minimum, two pictures will be taken from each survey plot: the first one will point from the downslope end up, and the second one will point from the upslope end down. Additional pictures of salient ecological features (e.g., particular grasses and flowering plants, bare spots lacking vegetation) will also be photographed, as needed. It is estimated that approximately 35 to 40 survey plots will be established across the MHWL in OU4. The GPS coordinates from the four corners of each survey plot will be documented.
- Each plot will be surveyed to identify the presence of obligate hydrophytic plants using the U.S. Army Corps of Engineers National Wetlands Plant List (USACE 2018). This online resource provides state-specific wetland plant lists, including for New York. The New York list will be used for this survey. Plants categorized as Obligate (OBL) wetland plants (e.g., American white water-lily, water clover, duckweed, American mana grass) are selected because they constitute perennial, vascular plants that always occur in standing water or saturated soil (USACE 2012) and reflect the presence of aquatic habitat. It is recognized that certain plants categorized as OBL, such as cat-tail, can be found in areas not containing continual water. For this reason, the use of the OBL category as defined in the Manual is considered a conservative surrogate for areas containing sediment associated with the Chemung River and Post Creek.

[1] https://waterdata.usgs.gov/nwis/monthly/?referred_module=sw&site_no=01529950&por_01529950_105507=1049814,00060,105507,1974-10,2019-12&format=html_table&date_format=YYYY-MM-DD&rdb_compression=file&submitted_form=parameter_selection_list

- If one or more OBL plants are identified within a survey plot, then the field biologist will carefully look to find any other OBL plants in that plot. If a total 50 percent or more of the surface area of the plot consists of OBL plants, then the substrate at that location will be classified as "sediment." Alternatively, if less than 50 percent of the surface area of the plot consists of OBL plants, then the substrate at that location will be classified as "soil." The 50 percent level is utilized as a conservative cutoff, given that some OBL plants, such as cat-tail, are not always found with continual water present in areas reflective of sediment associated with the Chemung River or Post Creek.
- If the substrate at a particular plot along the MHWL is determined to consist of soil based on vegetative characteristics, then the biologist will move downslope in 10-m increments toward the water in a straight line perpendicular to the flood control berm. The biologist will observe the vegetative species present, and specifically note the presence of OBL plant species. Upon identifying the presence of such plants, the biologist will establish a new 3 m wide by 10 m long survey plot perpendicular to the water and evaluate the plot with respect to the occurrence of OBL plants at 50 percent or more the surface area coverage. This new plot will be surveyed, photographed and surveyed with GPS as described earlier, and will be assigned as "soil" or "sediment", accordingly. The new estimated MHWL along that perpendicular transect will be located at the upslope end of the new survey plot. Of note, substrate will be defined as "soil" all the way downslope to the water's edge of the Chemung River or Post Creek if the number of OBL plants is less than 50 percent the surface area coverage.
- If the substrate at a particular plot along the estimated MHWL is determined to be sediment, then the biologist will move upslope in 10-m increments in a straight line perpendicular to the Chemung River or Post Creek. The goal is to identify another area where OBL plants may cover 50 percent or more of a survey plot. If no such area is identified upslope, then the upslope boundary of the previous plot would be retained as the sediment boundary at the estimated MHWL. If an additional upslope area contains OBL plants with the potential to cover 50 percent or more of a survey plot, then a new 3-m-wide by 10-m-long survey plot will be established and surveyed as described previously, and the new estimated MHWL along that perpendicular transect will be located at the downslope end of the new survey plot. This process will continue as far upslope toward the berm as needed.
- The goal of the survey is to differentiate between "soil" and "sediment" in OU4 in order to establish a boundary line for assessment of soil versus sediment. All substrate samples collected downslope of that line represent a conceptual approximation of sediment associated with the Chemung River or Post Creek, whereas all substrate samples collected upslope of that line are considered soil. The biologist will be aware of the potential presence of OBL plants at locations in OU4

that may occur above the estimated MHWL but are unrelated to aquatic habitat associated with the Chemung River or Post Creek. For example, a small local depression that holds water longer than the surrounding area may contain OBL plants. Such a feature will not be used to establish the estimated MHWL.

- The refined MHWL will be finalized at the completion of the survey, with concurrence from NYSDEC biologist.
- An appendix to the OU4 Site Characterization Report will include the supporting information to the finalization of the MHWL, including field notes and photographs.

References

Causton, D.R. 1988. *Introduction to Vegetation Analysis*. Unwin Hyman, London, England.

Kent, M., and P. Coker. 1992. *Vegetation Description and Analysis*. Belhaven Press, London, England.

NYSDEC. 1995. New York State Freshwater Wetlands Delineation Manual. July. Available at: http://www.dec.ny.gov/docs/wildlife_pdf/wdelman.pdf. New York State Department of Environmental Conservation.

NYSDEC. 2010. DER-10 / Technical Guidance for Site Investigation and Remediation. DEC Program Policy. New York State Department of Environmental Conservation, Division of Environmental Remediation. Issued May 3, 2010. Errata issued November 7, 2017.

NYSDEC. 2014. Screening and Assessment of Contaminated Sediment. Available at: http://www.dec.ny.gov/docs/fish_marine_pdf/screenasssedfin.pdf. New York State Department of Environmental Conservation, Division of Fish, Wildlife and Marine Resources, Bureau of Habitat. June 24.

USACE. 2012. National Wetland Plant List – Fact Sheet. Available at: https://www.lrl.usace.army.mil/Portals/64/docs/regulatory/Wetlands/WetlandPlantListFactSheet.pdf. U.S. Army Corps of Engineers, Headquarters – Regulatory Community of Practice.

USACE. 2018. NWPL – National Wetland Plant List home page. http://wetland-plants.usace.army.mil/nwpl_static/v34/home/home.html. U.S. Army Corps of Engineers.

USGS. 1995. Field Methods for Vegetation Mapping. USGS/NPS Vegetation Mapping Program. December. Available at: https://www1.usgs.gov/vip/standards/fieldmethodsrpt.pdf. U.S. Geological Survey.

Zonneveld, I.S. 1988. Landscape (ecosystem) and vegetation maps, their relation and purpose. pp. 481–486. In: *Vegetation Mapping*. A.W. Kuchler and I.S. Zonneveld (eds). Kluwer Academic Publishers, Boston, MA.