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PFAS PRELIMINARY SITE INVESTIGATION REPORT

CBJ – Juneau International Airport

1873 Shell Simmons Drive, Juneau, Alaska

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Prepared for:

City & Borough of Juneau
155 S. Seward Street
Juneau, AK 99801

Submitted to:

Alaska Department of Environmental Conservation
Division of Spill Prevention and Response
Contaminated Sites Program
410 Willoughby Ave., Ste. 303
P.O. Box 111800
Juneau, AK 99811

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ACRONYM LIST

Alaska Department of Conservation (ADEC)
Aqueous film-forming foams (AFFF)
Below ground surface (bgs)
City & Borough of Juneau (CBJ)
Cox Environmental Services (CES)
Direct Pushed Technology (DPT)
Dissolved oxygen (DO)
Granular activated carbon (GAC)
High density polyethylene (HDPE)
Investigation-Derived Wastes (IDW)
Lifetime health advisory (LHA)
Matrix spike/matrix spike duplicates (MS/MSDs)
Oil water separator (OW/S)
Oxygen reduction potential (ORP)
Per and Polyfluoroalkyl Substances (PFAS)
Perfluorobutanesulfonic acid (PFBS)
Perfluoroheptanoic acid (PFHpA)
Perfluorohexanesulfonic acid (PFHxS)
Perfluorononanoic acid (PFNA)
Perfluorooctane sulfonate (PFOS)
Perfluorooctanoic acid (PFOA)
Personal protective equipment (PPE)
Quality Assurance/ Quality Control (QA/QC)
Relative Percent Difference (RPD)
United States Environmental Protection Agency (USEPA)

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1 Introduction

Cox Environmental Services (CES) has been contracted by the City & Borough of Juneau (CBJ) to provide environmental services in support of characterization of Per and Polyfluoroalkyl Substances (PFAS) contamination. This Site Investigation Report presents the results for preliminary soil and groundwater sampling activities related to PFAS contamination at the Juneau International Airport site (JIA).

2 Site Description

The site is located at 1873 Shell Simmons Drive in Juneau, Alaska. The location of the site is depicted on *Figure 1. Site Location Map*. The features and details of the property are depicted on *Figure 2. 2013 Aerial Photograph* and *Figure 3. Site Plan* (Attachment A).

JIA is owned and operated by the CBJ. The Airport was originally developed by the United States government to support military Air Corps operations in Alaska. Prior to World War II, the area was served by a limited number of small aircraft, mostly float planes. The paved runway at the Airport was constructed in 1942. Following the war, Pan American Airlines and Pacific Northern Airlines established service to Juneau from Seattle and Anchorage. The original terminal was constructed in 1948. In 1953, the Airport was transferred from U.S. government ownership to the City of Juneau. The first of two major terminal expansions took place in 1957, and the second expansion, resulting in the Airport's present configuration, took place in 1984. The Airport has undergone other modifications as well. In 1961, the runway was extended to accommodate jet aircraft operations in Alaska. In 1989, a full-length parallel taxiway was constructed to connect both ends of the runway to the aircraft parking apron and passenger terminal area. Other facility improvements have taken place periodically, most recently for additional aircraft parking and hangar spaces.

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JIA is owned and operated by the CBJ. The Airport was originally developed by the United States government to support military Air Corps operations in Alaska. Prior to World War II, the area was served by a limited number of small aircraft, mostly float planes. The paved runway at the Airport was constructed in 1942. Following the war, Pan American Airlines and Pacific Northern Airlines established service to Juneau from Seattle and Anchorage. The original terminal was constructed in 1948. In 1953, the Airport was transferred from U.S. government ownership to the City of Juneau. The first of two major

terminal expansions took place in 1957, and the second expansion, resulting in the Airport's present configuration, took place in 1984. The Airport has undergone other modifications as well. In 1961, the runway was extended to accommodate jet aircraft operations in Alaska. In 1989, a full-length parallel taxiway was constructed to connect both ends of the runway to the aircraft parking apron and passenger terminal area. Other facility improvements have taken place periodically, most recently for additional aircraft parking and hangar spaces.

3.1 Climate

Juneau is near the northern end of a temperate rain forest found on the North Pacific Coast from San Francisco to Anchorage. Juneau weather is characterized by a North Pacific maritime climate with frequent storms and abundant precipitation. The mean summer high temperature is 62° F, and the mean winter high temperature is 33° F. There are approximately 150 frost-free days annually.

3.2 Hydrology

The JIA is located on the Mendenhall River delta. Material excavated from the Float Plane Pond was used in the original airport construction (Adamus 1987) in the late 1930s and early 1940s, and a wide variety of other sources were used as fill to develop the large elevated surface that the JIA now occupies.

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The JIA is bordered by the Mendenhall River on the west, the Mendenhall Wetlands Refuge to the south and east, and industrial and commercial land to the north. A dike in the western and southern portions of the airport property protects the Float Plane Pond and other assets from the Mendenhall River to the west and from the Gastineau Channel to the south and southeast. The Gastineau Channel inundates the refuge daily at high tide.

Duck Creek enters the airport property from the northwest, through a culvert under Berners Avenue. Duck Creek bends southwest through an undeveloped parcel of land in the northwest corner of the airport property. Duck Creek leaves the site via a culvert that passes under the dike to the west and discharges to the Mendenhall River approximately 1,500 feet later. The creek has been channelized in several reaches through infrastructure development activities. The floodplain is constricted in many locations.

Jordan Creek enters the airport property from the north approximately 1,400 feet east of Duck Creek. Jordan Creek crosses Yandukin Drive and meanders for approximately 1,300 feet before crossing underneath Crest Street through a culvert. Jordan Creek is channelized along portions of the reach below Yandukin Drive, and the floodplain is especially constricted below Crest Street. The channel bends sharply as it travels through the airport property. The creek passes through long culverts under the taxiway and then the runway prior to leaving the airport property and entering the refuge.

Vegetated ditches that drain most of the runways and taxiways discharge stormwater to Duck Creek and Jordan Creek. High tides create backwater conditions on Jordan Creek that can cause ponding in these ditches.

The Float Plane Pond is surrounded by the runway to the north, the Mendenhall River to the west, the dike to the south, and the mouth of Jordan Creek to the east. A dike separates the Float Plane Pond from tidal wetlands and the Gastineau Channel. The Float Plane Pond is approximately 5,300 feet long by 430 feet wide, with an average depth of four to five feet. A 30-foot deep pocket of water is located in the south end of the pond. Several sloughs and side channels extend from the main body of the pond into the wooded area to the south. The total surface area of the Float Plane Pond is approximately 80 acres, including sloughs and side channels. The water level of the pond is controlled by a tide gate at the west end of the pond. During high tide conditions, brackish water from the Mendenhall River enters the pond through this structure.

3.3 General Geology and Soils

The JIA is located on the northeast side of Gastineau Channel, within the Mendenhall River Basin, which extends from the Coast Mountains of southeast Alaska. Several studies have described the geology of the Mendenhall Valley (Alcorn and Hogan 1995, Barnwell and Boning 1968, Hicks and Shofnos 1965, Motyka 1988). The underlying bedrock is composed of tightly consolidated sedimentary (slate, greywacke, and sandstone), igneous (extruded volcanics), and metamorphic rocks (greenstone and schist) that are relatively impervious to moisture. The surficial geology in the area around the Gastineau Channel, the JIA, and Mendenhall Valley includes glaciomarine deposits of the Gastineau Channel Formation, overlain by glacial outwash deposits. The outwash deposits range in thickness from 10 feet to 100 feet. They are comprised of sand-size to cobble-size rocks that have been overlain in some small areas, mostly down the middle of the valley, by muskeg or plant debris in various stages of decay. Moraine deposits composed of loose till and unsorted gravelly sand are found in the upper valley. Farther down the valley, beach deposits and glaciomarine deposits from the Gastineau Channel Formation characterize most of the Gastineau Channel.

The JIA property consists of the BeA, CoA, and LeA soil-mapping units, with BeA the predominant mapping unit on the JIA property (USDA 1974). BeA is excessively drained, very gravelly sand with 0% to 3% slopes. This soil is found on nearly level alluvial plains and terraces, along with spots of wet, sandy soils. This soil rarely floods. However, in a few low-lying areas near the coast and adjacent to streams, inundation may occur when tides or streams are exceptionally high. A small portion of the northwest JIA property consists of the CoA soil-mapping unit. This soil is a poorly drained silt loam found on low-lying, nearly level, alluvial plains. The angles of slopes on which this soil is found range from 0% to 3%. In most

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places, this soil is susceptible to occasional overflow from freshwater streams, and in a few places it may be inundated by exceptionally high tides. This soil unit may include spots of very poorly drained shallow peat soils. A small section on the northern edge of the JIA property consists of the LeA soil-mapping unit. This mapping unit includes areas of small streams. The soil is a very poorly drained silt loam found on slight depressions in broad stream valleys; the slope ranges from 0% to 3% and is almost always nearly level. This soil is susceptible to occasional flooding.

3.4 Groundwater

The Mendenhall Valley contains two aquifers (Barnwell and Boning 1968). The upper aquifer lies within the unconfined sediments of silt, sand, and gravel at a depth of 3 to 15 feet below the ground surface. The thickness of the upper aquifer ranges from 0 to 300 feet (Osgood 1990). Mendenhall Lake is a major source of recharge for the upper aquifer. The lower aquifer is separated from and confined by a layer of bedrock below the upper aquifer. The water table flows southwesterly through the valley towards the Mendenhall River.

The JIA is located on the Mendenhall River delta, and the runway and associated taxiways were built up by fills of sand and gravel, mostly from the excavation of the Float Plane Pond. The porous nature of the geology under the JIA, possibly including the old stream channels from Duck Creek and Jordan Creek, likely facilitates groundwater interaction with surface water.

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4 Previous Investigations

No previous investigations related to PFAS have been conducted at the site.

5 PFAS Information

PFAS are a group of synthetic chemicals that have been in use since the 1940s. PFAS are found in a wide array of consumer and industrial products. PFAS manufacturing and processing facilities, facilities using PFAS in production of other products, airports, and military installations are some of the contributors of PFAS releases into the air, soil, and water. Due to their widespread use and persistence in the environment, most people in the United States have been exposed to PFAS. There is evidence that continued exposure above specific levels to certain PFAS may lead to adverse health effects (USEPA 2016a, 2016b, ATSDR 2018a).

In Alaska, spills or releases of PFAS into the environment are primarily associated with the use of aqueous film-forming foams (AFFF) during firefighting or fire training activities. PFAS compounds of concern where AFFF has been used include perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). Although these two compounds are the most studied, a growing body of research indicates

additional PFAS compounds may have similar health or environmental effects and may be co-contaminants. In 2016, The ADEC published cleanup levels for PFOS and PFOA and the United States Environmental Protection Agency (USEPA) issued lifetime health advisory (LHA) levels for these compounds in drinking water. In 2018, ADEC set action levels for six PFAS compounds, including PFOS and PFOA. On April 9, 2019, ADEC published a revised Technical Memorandum on Action Levels for PFAS that supersedes the 2018 action levels memorandum and aligns the action levels with EPA's LHA levels for PFOS and PFOA. Action levels serve as thresholds for determining when responsible parties need to provide water treatment or alternative water sources for impacted water supplies.

Because PFAS are persistent in the environment and soluble in water, large plumes of groundwater contamination can form where these compounds have been released. When releases occur in areas served by private or public drinking water wells, the well water is susceptible to contamination. When PFAS contamination is found in the environment, the responsible party must evaluate the extent of the contamination in the soil and groundwater, determine whether and to what extent drinking water supplies are impacted, provide treatment or alternative water if action levels are exceeded, and begin cleanup with ADEC's oversight. The responsible party is typically the entity that caused the release or the landowner where the release occurred.

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Title 14 Code of Federal Regulation (CFR) Part 139 requires airport operators to maintain their aircraft rescue and firefighting (ARFF) vehicles and their fire suppression operating systems. JIA is a Part 139 Airport. Such systems, including the foam proportioning system and discharge functions, must be able to operate properly in an emergency situation. To help ensure their operability, the Federal Aviation Administration (FAA) recommends vehicle system testing intervals occur within 6 months of the airport's periodic airport certification safety inspection. Currently, all certificated Part 139 airports are required to use foams that meet military specifications (MIL-PRF-24385). The growing concern over the use and discharge of AFFF at airports has led to the inclusion of a mandate within the FAA Reauthorization Act of 2018 (enacted October 5, 2018), directing the FAA to stop requiring the use of fluorinated foam no later than three years from the date of enactment (October 4, 2021).

The airport currently uses CHEMGUARD C306-MS 3% AFFF, the formulation contains short-chain, C-6 fluorochemicals manufactured using a telomer-based process. The telomer process produces no PFOS, and these C-6 materials do not breakdown to yield PFOA. Historical releases of PFAS from AFFF during historical training activities are believed to have taken place in five locations at the airport, actual quantities of AFFF or dates of discharges at the site are not documented. The historical release locations are depicted on *Figure 5 Potential AFFF Discharge Areas.*

6 Environmental Investigation

The soil and groundwater sampling activities were performed in general accordance with following regulations & documents:

- 18 AAC 75: Oil and Other Hazardous Substances Pollution Control
- ADEC's Field Sampling Guidance (ADEC August 2017)
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for PFAS, (Interstate Technology Regulatory Council's (ITRCs) March 2018)
- Bottle Selection and other Sampling Considerations When Sampling for PFAS (USDOD EDQW 2017b)
- Interim Guideline on the Assessment and Management of PFAS, Contaminated Sites Guidelines, (Government of Western Australia, Department of Environment Regulation 2016)
- Alpha Analytical EPA 537 (PFAS) Field Sampling Guidelines

The environmental investigation consisted of:

- Installation of six (6) soil borings and collection of one subsurface soil sample from each boring.
- Installation of six (6) groundwater monitoring wells to evaluate potential impacts to groundwater.
- Collection of soil and groundwater samples for laboratory analysis.

The soil borings and monitoring well locations are depicted on *Figure 4. Site Plan with Soil Borings and Groundwater Monitoring Well Locations.*

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7 Soil Sampling & Laboratory Analyses

The soil borings were advanced with a rotary auger drill rig with a 24-inch stainless steel split spoon sampler. One soil sample was collected at each soil boring from near the soil groundwater interface. CES personnel used disposable spoons to collect soil samples. Prior to handling any soil, CES personnel donned a new pair of disposable nitrile gloves which were interchanged prior to collection of each soil sample. Soil samples to be analyzed for PFAS were placed into laboratory certified unpreserved 4-oz high density polyethylene (HDPE) jars. All sample jars were labeled with the project name, sample identification number, date/time of sample collection, preservative, analysis requested, and sampler's initials and placed in zip-lock bags. The samples were kept in a sample cooler with double bagged ice (at 4°C ± 2°C) pending delivery to the contract laboratory. Laboratory reports and analytical checklists are included in Attachment B.

8 Groundwater Sampling & Laboratory Analyses

The groundwater monitoring wells were also installed using a rotary auger drill rig. The installation of the groundwater monitoring wells was performed in general accordance with ADEC's Monitoring Well

Guidance, September 2013. The groundwater monitoring wells were completed with Geoprobe 2.0 in. Slim Prepacks (2.0-in diameter, prepacked with 20/40 sand, constructed from Schedule 40 PVC with a 5-ft screen section of 0.010 inch slotted screen, riser and threaded end caps). The outer filter pack made from 10/20 sand was added to 1-ft above the top of the screen, bentonite chips were added to surface. The monitoring wells were completed with aboveground monuments. Groundwater well installation was in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling, Attachment D). Preliminary construction logs for the monitoring well installations are included in Attachment C. The logs will be finalized after the wells are surveyed.

The groundwater monitoring wells were developed and then after development, CES personnel used a peristaltic pump and disposable PFAS-free silicone and HDPE tubing to purge the well prior to sampling. When purging monitoring wells prior to sampling, CES removed at least three casing volumes. The groundwater monitoring wells were developed and purged in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling, Attachment D).

After purging, CES personnel used a peristaltic pump and disposable PFAS-free silicone and HDPE tubing to collect groundwater samples. Prior to handling any groundwater, CES personnel donned a new pair of disposable nitrile gloves which were interchanged prior to collection of each groundwater sample. Each groundwater sample for PFAS was collected into two laboratory certified unpreserved 250 ml-HDPE containers. All containers were labeled with the project name, sample identification number, date/time of sample collection, preservative, analysis requested, and sampler's initials and placed in zip-lock bags. The samples were kept in a sample cooler with double bagged ice (at 4°C ± 2°C) pending delivery to the contract laboratory. The groundwater monitoring wells were sampled in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling, Attachment D).

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9 Analytical Methods

Soil and groundwater samples were analyzed by our contract laboratory, Test America, Inc. Samples were analyzed for:

- The full suite of PFAS compounds using Modified Method 537
  - 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
  - 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
  - ADONA
  - DONA
  - HFPO-DA (GenX)

- N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)
- N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)
- Perfluorobutanesulfonic acid (PFBS)
- Perfluorodecanoic acid (PFDA)
- Perfluorododecanoic acid (PFDoA)
- Perfluoroheptanoic acid (PFHpA)
- Perfluorohexanesulfonic acid (PFHxS)
- Perfluorohexanoic acid (PFHxA)
- Perfluorononanoic acid (PFNA)
- Perfluorooctanesulfonic acid (PFOS)
- Perfluorooctanoic acid (PFOA)
- Perfluorotetradecanoic acid (PFTeA)
- Perfluorotridecanoic acid (PFTriA)
- Perfluoroundecanoic acid (PFUnA)

Analytical data validation was performed by CES by filling out the ADEC Laboratory Data Review Checklist for each laboratory data deliverable. The laboratory analysis reports and checklists are included in Attachment B.

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10 ADEC Cleanup Levels

According to the October 2, 2019 revised Technical Memorandum, Action Levels for PFAS in Water and Guidance on Sampling Groundwater and Drinking Water, in order to align state actions to the recently announced USEPA plans, **ADEC will use the USEPA LHA (PFOS+PFOA above 70 ng/L) as the Action Level. Any new testing for PFAS will report the full suite of PFAS compounds analyzed by the appropriate EPA Method.**

For the purposes of this investigation, soil analytical data will be compared to the over 40-inch cleanup levels in 18 AAC 75.341 Table B1. Method Two – Soil Cleanup Levels Table. **The PFOS Human Health cleanup level is 1,300 µg/kg and the Migration to Groundwater Cleanup level is 3 µg/kg. The PFAS Human Health cleanup level is 1,300 µg/kg and the Migration to Groundwater Cleanup level is 1.7 µg/kg.**

Groundwater analytical data will be compared to ADEC groundwater cleanup levels in 18 AAC 75.341 Table C. Groundwater Cleanup Levels. **The PFOS Groundwater Cleanup level is 400 ng/L and the PFAS Groundwater Cleanup levels is 400 ng/L. Groundwater analytical data will also be compared to the USEPA LHA (PFOS+PFOA above 70 ng/L).**

11 Soil Analytical Results

Table 1. Summary of Soil Data – November 2019 in Attachment A summarizes the soil sampling results relative to ADEC cleanup levels.

PFOS was detected at a concentration of 1.2 µg/kg in MW-1; not detected in MW-2, MW-3, MW-4 or MW-5; and detected at a concentration of 31 µg/kg in MW-6. PFOA was not detected in MW-1, MW-2, MW-3, MW-4, or MW-5; and detected at a concentration of 0.44 µg/kg in MW-6. One of the two detected concentrations exceed the ADEC Method 2 Migration to Groundwater Cleanup Level of 3 µg/kg. All of the detected concentrations of PFAS contaminants are less than the ADEC Method 2 Human Health Cleanup level of 1,300 µg/kg.

Other PFAS constituents that do not have corresponding cleanup levels were also detected in one of the six soil samples. PFHpA was not detected in MW-1, MW-2, MW-3, MW-4, and MW-5; detected at a concentration of 0.21 µg/kg in MW-6. PFHxS was not detected in MW-1, MW-2, MW-3, MW-4, and MW-5; detected at a concentration of 1.2 µg/kg in MW-6.

12 Groundwater Analytical Results

Table 2. Summary of Groundwater Data – January 2020 in Attachment A summarizes the groundwater sampling results relative to ADEC and USEPA cleanup levels.

PFOS was detected in five of the five samples at concentrations ranging from 3.4 ng/L to 750 ng/L. PFOS was detected at a concentration of 340 ng/L in MW-1; 750 ng/L in MW-3; 67 ng/L in MW-4; 3.4 ng/L in MW-5; and 540 ng/L in MW-6. Two of the five detected concentrations exceed the ADEC Groundwater Cleanup Level of 400 ng/L.

PFOA was detected in four of the five samples at concentrations ranging from 29 ng/L to 410 ng/L. PFOA was detected at a concentration of 29 ng/L in MW-1; 34 ng/L in MW-3; 410 ng/L in MW-4; not detected in MW-5; and 40 ng/L in MW-6. One of the detected concentrations exceed the ADEC Groundwater Cleanup Level of 400 ng/L.

The calculated concentrations of PFOS+PFOA are 369 ng/L in MW-1; 784 ng/L in MW-3; 477 ng/L in MW-4; 3.4 ng/L in MW-5; and 580 ng/L in MW-6. Four of the five calculated concentrations of PFOS+PFOA exceed the USEPA LHA of 70 ng/L.

Other PFAS constituents that do not have corresponding cleanup levels were also detected in five of the five samples. PFBS was detected at a concentration of 27 ng/L in MW-1; not detected in MW-3; 670 ng/L

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in MW-4; not detected in MW-5; and 16 ng/L in MW-6. PFDA was not detected in MW-1; detected at a concentration of 2.4 ng/L in MW-3; not detected in MW-4; not detected in MW-5; and not detected in MW-6. PFHpA was detected at a concentration of 37 ng/L in MW-1; 24 ng/L in MW-3; 390 ng/L in MW-4; not detected in MW-5; and 41 ng/L in MW-6. PFHxS was detected at a concentration of 220 ng/L in MW-1; 28 ng/L in MW-3; 1,900 ng/L in MW-4; 1.8 ng/L in MW-5; and 110 ng/L in MW-6. PFHxA was detected at a concentration of 130 ng/L in MW-1; 35 ng/L in MW-3; 1,500 ng/L in MW-4; not detected in MW-5; and 73 ng/L in MW-6. PFNA was detected at a concentration of 3.1 ng/L in MW-1; 10 ng/L in MW-3; 5.0 ng/L in MW-4; not detected in MW-5; and 6.5 ng/L MW-6.

MW-2 was non-productive during sampling and appears to be affected by precipitation and/or tidal fluctuations.

13 Decontamination Procedures

Field sampling equipment, including oil/water interface meters, direct-push tooling, and other non-dedicated equipment used at each sample location were decontaminated between use. CES used Alconox detergent which is PFAS-free and CBJ public water during decontamination of non-dedicated sampling equipment and drill tooling. The CBJ public water was previously sampled during the Hagevig Fire Training Center investigation and documented to be PFAS-free. The equipment decontamination was in accordance with CES Standard Operating Procedure SP-04 (SP-04 Equipment Decontamination Procedures, Attachment D).

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14 Investigation-Derived Wastes (IDW)

During the site investigation, CES generated potentially contaminated IDW that included the following:

- Used personal protective equipment (PPE)
- Disposable sampling equipment
- Decontamination fluids
- Soil cuttings from soil borings
- Purged groundwater

Used PPE and disposable sampling equipment was double bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and were sent to the local landfill.

Decontamination fluids (residual contaminants, water with non-phosphate detergent) and purged groundwater (contaminants, water) were containerized and then filtered on-site with a portable granular

activated carbon (GAC) filter system and reapplied to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies.

Soil cuttings from soil borings were containerized in a 55-gallon, DOT approved, steel drum labeled as to type of waste (soil), the source location, and date. Soil IDW will be transported to NRC Alaska for disposal and treatment after an ADEC Soil Transport Form is completed and approved by ADEC.

15 Quality Assurance/Quality Control

Measures of quality include the appropriateness and accuracy of the sample collection; adherence to sample handling protocols; the quality and appropriateness of the laboratory analysis; and the representativeness of the data with respect to the study objectives. Modified Method 537 requires a field reagent blank be collected at each site where field samples are collected. Trip blanks are not applicable to Method 537 and additionally with the use of an isotope dilution method, Modified 537, the inclusion of matrix spike/matrix spike duplicates (MS/MSDs) is unnecessary.

Analytical data validation was performed by CES by filling out the ADEC Laboratory Data Review Checklist for each laboratory data deliverable. The laboratory analysis reports and checklists are included in Attachment B. All analytical data collected are usable for the purposes of this investigation, precision, accuracy, representativeness, comparability, and completeness are deemed to be within acceptable tolerance. The following table lists the minimum field QC samples, applicability, and allowable tolerance.

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Minimum Field QC Samples	Applicability	Allowable Tolerance
Field Duplicate (Minimum of one per every 10 field samples for each matrix sampled, for each day in field, for each target analyte, minimum of one)	All soil & water samples collected on the same day	Relative percent differences (RPD) less than: 30% water, 50% soil
Decontamination or Equipment Blank (One per set of 20 similar samples, minimum of one)	Per project specifications, to include soil sampling spoons, groundwater sampling tubing, and/or direct push tooling.	Less than the practical quantitation limit
Field Reagent Blank (One per set of 20, minimum of one)	Per project specifications and per Modified Method 537	Less than the practical quantitation limit
Temperature Blank	Per project specifications, one per cooler	4°C+/-2°C

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15.1 Field Duplicate Samples

CES collected one field duplicate for each matrix sampled and for each target analyte. Field duplicates were collected from a location of suspected contamination, and duplicate soil and water samples were collected in the same manner and at the same time and location as the primary sample. Field duplicates were submitted as blind samples to the laboratory for analysis, given unique sample numbers (or names) and sample collection time, and adequately documented in the field record or log book. Field duplicate results were used to calculate and report a precision value for field sampling quality control. The RPD for PFAS in the soil duplicate was within the allowable tolerance of 50% and the RPD for the groundwater duplicate was within the allowable tolerance of 30%.

15.2 Equipment Blanks

CES collected three equipment blanks to trace sources of artificially introduced contamination. Equipment blanks consisted of PFAS free water (reagent-grade) supplied by the laboratory poured over or through decontaminated field sampling equipment prior to the collection of environmental samples. Equipment blanks were given unique sample numbers (or names) and sample collection time, and adequately documented in the field record or log book. Equipment blanks were not submitted blind to the laboratory. No PFAS constituents were detected in the equipment blank samples, all results were less the practical quantification limit.

15.3 Field Blanks (Reagent Blanks)

CES collected one field blank (reagent blank) per matrix to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected. Field blanks (reagent blanks) consisted of PFAS free water (reagent-grade) supplied by the laboratory, transported to the sampling site, handled like an environmental sample (exposed to sampling equipment/materials), and returned to the laboratory for analysis. The field blanks were created by attaching 10 feet of new tubing to the peristaltic pump head and withdrawing enough volume of laboratory-supplied water to fill two sample containers and by pouring water over the soil spoons to fill two sample containers. Field blanks (reagent blanks) were submitted blind to the laboratory. No PFAS constituents were detected in the field blank samples, all results were less the practical quantification limit.

15.4 Temperature Blank

A temperature blank accompanied the coolers transporting samples to the laboratory, the laboratory measured the temperature of the temperature blank and compared the results against the QA/QC requirement of 4 ± 2 °C during sample receipt procedures. The soil samples were received on 11/20/2019 10:10 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 6.1°C. The groundwater samples were received on 1/7/2020 11:38 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 5.2°C.

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16 Summary and Recommendations

This SIR presents the results of preliminary soil and groundwater sampling activities at the Juneau International Airport in Juneau, Alaska.

The soil investigation included the collection of six soil samples from six borings.

- PFOS was detected in two of the six samples at concentrations ranging from 1.2 µg/kg to 31 µg/kg. One of the six detected concentrations (MW-6) exceeds the ADEC Method 2 Migration to Groundwater Cleanup Level of 3 µg/kg. All of the detected concentrations of PFOS are less than the ADEC Method 2 Human Health Cleanup level of 1,300 µg/kg.
- PFOA was detected in one of the six samples (MW-6) at a concentration of 0.44 µg/kg. The detected concentration is below the ADEC Method 2 Migration to Groundwater Cleanup Level of 1.7 µg/kg and less than the ADEC Method 2 Human Health Cleanup level of 1,300 µg/kg.

The groundwater investigation included the collection of five groundwater samples from five monitoring wells.

- PFOS was detected in five of the five samples at concentrations ranging from 3.4 ng/L to 750 ng/L. Two of the five detected concentrations (MW-3 and MW-6) exceed the ADEC Groundwater Cleanup Level of 400 ng/L.
- PFOA was detected in four of the five samples at concentrations ranging from 29 ng/L to 410 ng/L. One of the of the detected concentrations (MW-4) exceeds the ADEC Groundwater Cleanup Level of 400 ng/L.
- Four of the five calculated concentrations of PFOS+PFOA (MW-1, 369 ng/L; MW-3, 784 ng/L; MW-4, 477 ng/L; and MW-6 580 ng/L) exceed the USEPA LHA of 70 ng/L.

Based on the results of the preliminary soil and groundwater investigation, CES recommends the following:

- CBJ continue investigation of PFAS in soil and groundwater at the site and off-site surrounding properties to the south of the JIA to continue to work towards delineating the vertical and horizontal extent of PFAS contamination in soil and groundwater.
    - Additional soil borings and groundwater wells should be placed surrounding MW-6 to delineate the extent of PFAS contamination in soil and groundwater. MW-6 is located southwest of the Capital City Fire Rescue ARFF Station.
    - PFAS contamination was detected in MW-3 and MW-4, additional groundwater wells should be installed to the south of the JIA to delineate the extent of off-site groundwater contamination.
    - MW-2 was non-productive during sampling and appears to be affected by precipitation and/or tidal fluctuations. The well should be re-drilled and set with a 10' screen to allow for sampling during all conditions.
- CBJ should establish a quarterly groundwater sampling program to begin to establish a trend in groundwater concentrations.
    - Now that PFAS has been detected on-site, monitoring wells MW-10 and MW-4 which were installed during previous on-site investigations should be added to the well network for sampling.
- The groundwater monitoring wells installed during this investigation should be surveyed so that elevation data can be utilized to evaluate groundwater flow direction.
- CBJ public water service is available to properties throughout the project area. CBJ should conduct a door to door and/or letter based private well search to confirm whether there are private wells still in service within the project area. If wells are discovered CES recommends CBJ make arrangements to sample the wells. The ADEC will require the CBJ to provide potable water if wells are discovered to be contaminated and the property in not already connected to CBJ public water until public service can be provided.

DRAFT

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DRAFT

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ATTACHMENT A

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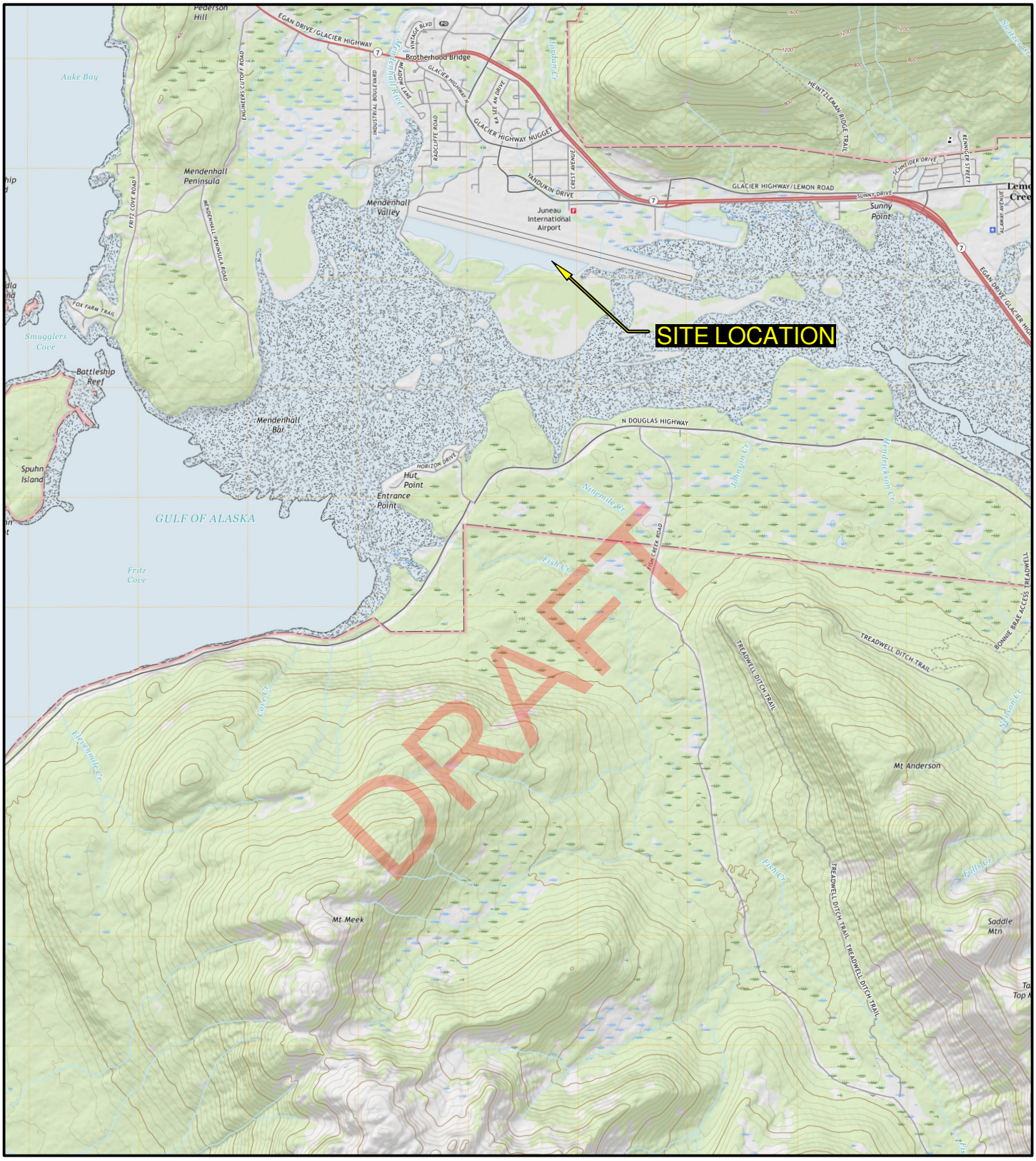


COX environmental services

712 W 12th Street Juneau, Alaska 99801 907.586.4447 www.coxenv.com

Prepared for: City & Borough of Juneau

FIGURE 1. SITE LOCATION MAP

Juneau International Airport
1873 Shell Simons Drive
Juneau, AK

Date:7.18.2019

COX environmental services
712 W 12th Street Juneau, Alaska 99801 907.586.4447 www.coxenv.com

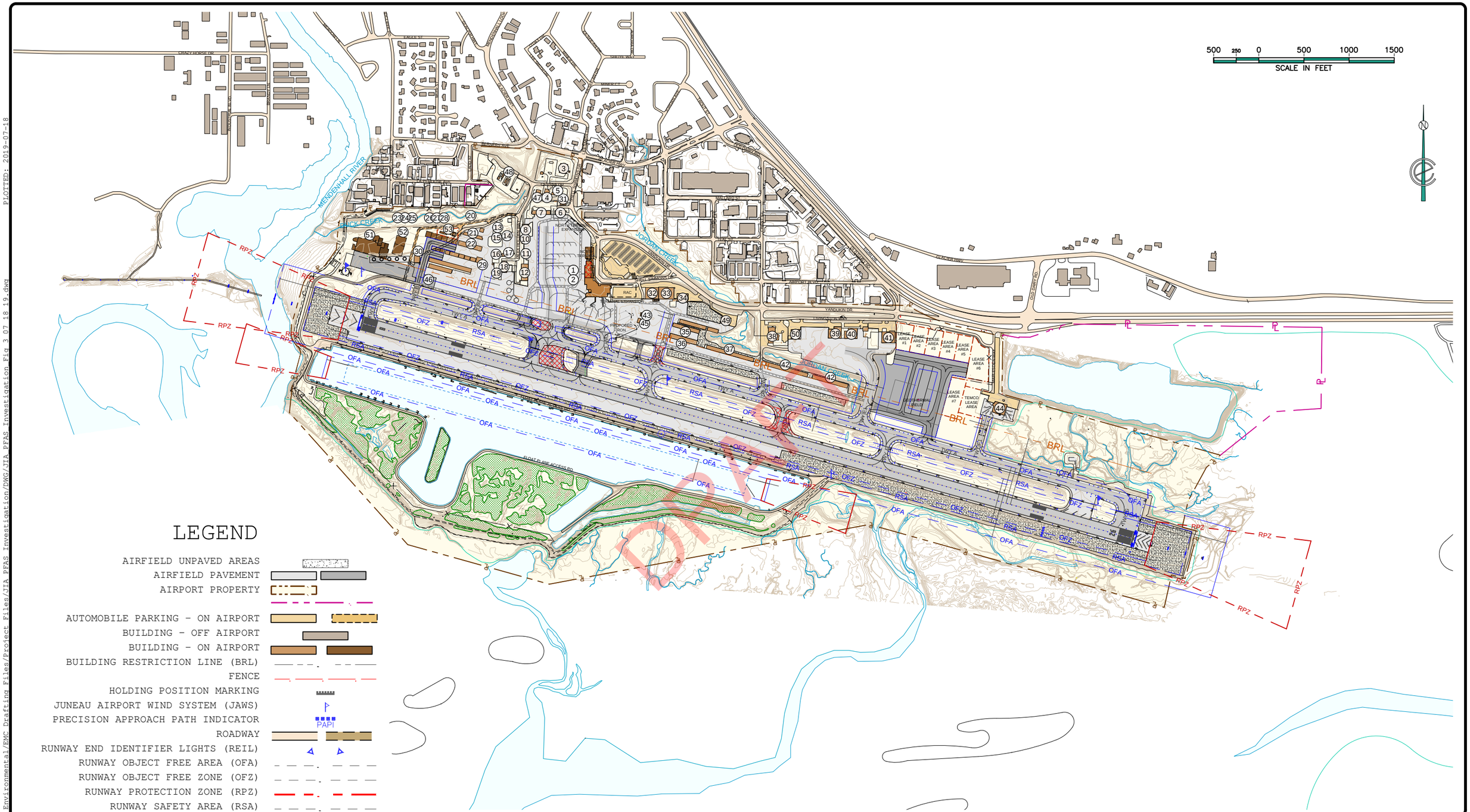
2013 Aerial Photograph

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE
DRAWN emc
CHECKED jmc
DATE 7/18/2019

Fig 2

FILE: /home/iolo/Cox Environmental/EMC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 2 07 18 19.dwg PLOTTED: 2019-07-18



LEGEND

- AIRFIELD UNPAVED AREAS
- AIRFIELD PAVEMENT
- AIRPORT PROPERTY
- AUTOMOBILE PARKING – ON AIRPORT
- BUILDING – OFF AIRPORT
- BUILDING – ON AIRPORT
- BUILDING RESTRICTION LINE (BRL)
- FENCE
- HOLDING POSITION MARKING
- JUNEAU AIRPORT WIND SYSTEM (JAWS)
- PRECISION APPROACH PATH INDICATOR (PAPI)
- ROADWAY
- RUNWAY END IDENTIFIER LIGHTS (REIL)
- RUNWAY OBJECT FREE AREA (OFA)
- RUNWAY OBJECT FREE ZONE (OFZ)
- RUNWAY PROTECTION ZONE (RPZ)
- RUNWAY SAFETY AREA (RSA)
- TAXIWAY OBJECT FREE AREA (TOFA)
- TO BE REMOVED
- TOPOGRAPHIC CONTOUR
- TREES
- VISUAL APPROACH SLOPE INDICATOR (VASI)
- WIND SOCK

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Site Plan

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE 1"=1000'
DRAWN jmc
CHECKED jmc
DATE 7/18/2019

Fig 3

FILE: /home/dojo/Cox Environmental/ENC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 3 07 18 19.dwg
PLOTTED: 2019-07-18

#	DESCRIPTION
1	TERMINAL BUILDING
2	AIRPORT TRAFFIC CONTROL TOWER (ATCT)
3	AFFSS/SACOM
4	J/D TELEPHONE
5	LEASED TO HERTZ
6	AERO SERVICES (SUBLEASE: FEDEX) R&L LEASING
7	AIRFIELD MAINTENANCE FACILITY
8	LAB FLYING
10	HEIMBIGNER
11	ALASKA AIRLINES
12	ALASKA AIRLINES
13	PRIVATE HANGAR
14	FISH AND WILDLIFE
15	3 PRIVATE T-HANGARS
16	DUANE PACKER (SUBLEASE: UNITED PARCEL SERVICE)
17	R&L LEASING
18	AERO SERVICES
19	AERO SERVICES
20	PRIVATE HANGAR
21	T-HANGAR (7 UNITS)
22	T-HANGAR (12 UNITS)
23	PRIVATE HANGAR
24	PRIVATE HANGAR
25	PRIVATE HANGAR
26	PRIVATE HANGAR
27	PRIVATE HANGAR
28	PRIVATE HANGAR
29	PRIVATE HANGAR
30	CIVIL AIR PATROL
31	DELTA WESTERN
32	SAND STORAGE [POTENTIAL ATCT LOCATION]
33	LOKEN AVIATION
34	WARD AIR (RED LEASING)
35	BLOCK 'L' EXECUTIVE HANGARS (5 UNITS)
36	BLOCK 'M' T-HANGARS (14 UNITS)
37	BLOCK 'N' T-HANGARS (9 UNITS)
38	AIRCRAFT RESCUE AND FIRE FIGHTING (ARFF)
39	SILVER BAY LOGGING
40	ALASKA NATIONAL GUARD
41	WINGS OF ALASKA
42	BLOCK 'O' EXECUTIVE HANGARS (14 UNITS)
43	COASTAL FUEL
44	TEMSCO HELICOPTERS
45	COASTAL HELICOPTERS
46	BLOCK 'I' HANGARS
47	R&L LEASING
48	FUEL FARM
49	ALASKA SEAPLANES HANGAR
50	GLACIER AVIATION (AIRLIFT NORTHWEST) HANGAR
51	FUTURE SNOW REMOVAL EQUIPMENT BUILDING (SREB)
52	FUTURE SAND CHEMICAL BUILDING
53	FUTURE FUEL FACILITY

DRAFT

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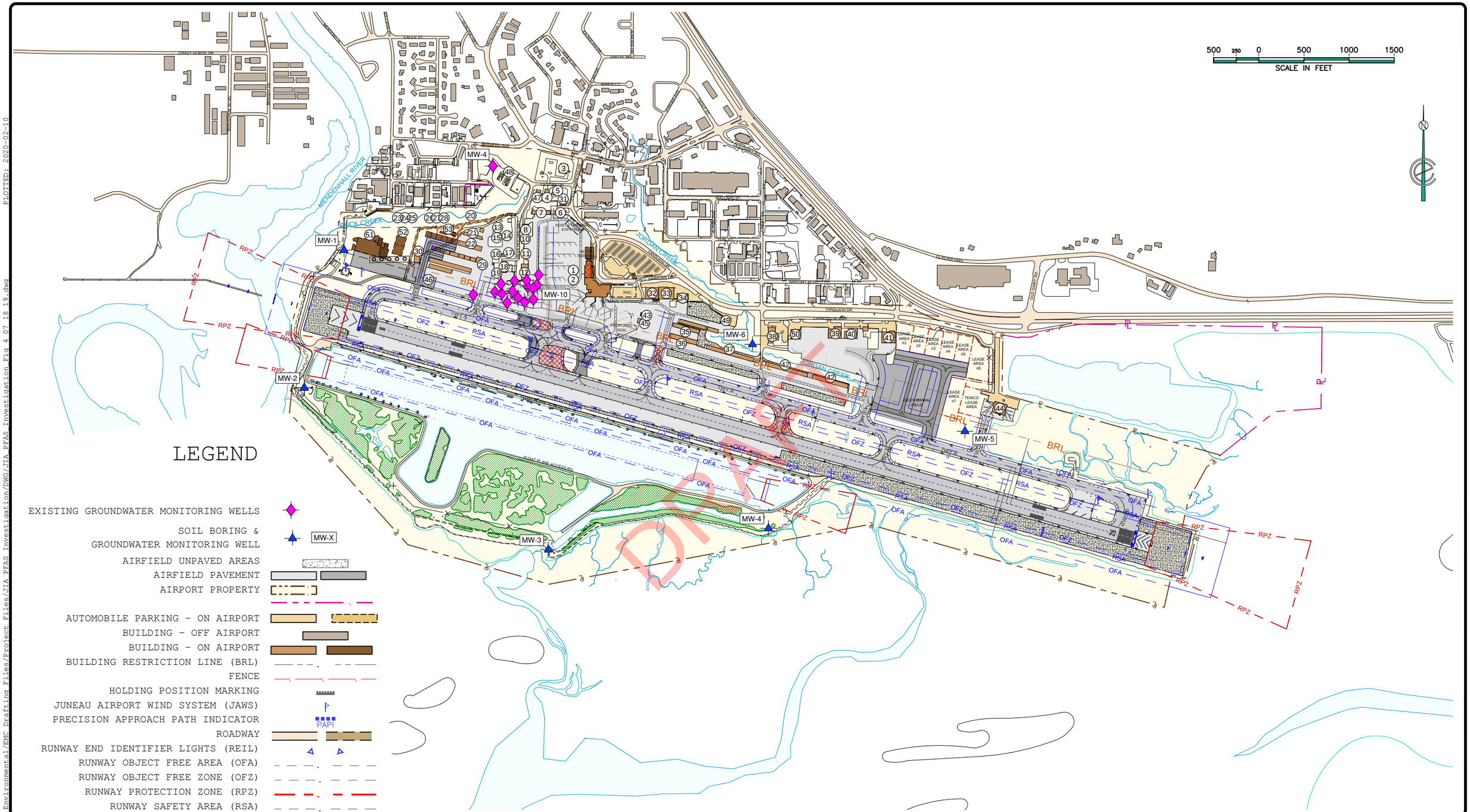
AIRPORT FACILITIES

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA



LEGEND

- EXISTING GROUNDWATER MONITORING WELLS
- SOIL BORING & GROUNDWATER MONITORING WELL (MW-X)
- AIRFIELD UNPAVED AREAS
- AIRFIELD PAVEMENT
- AIRPORT PROPERTY
- AUTOMOBILE PARKING – ON AIRPORT
- BUILDING – OFF AIRPORT
- BUILDING – ON AIRPORT
- BUILDING RESTRICTION LINE (BRL)
- FENCE
- HOLDING POSITION MARKING
- JUNEAU AIRPORT WIND SYSTEM (JAWS)
- PRECISION APPROACH PATH INDICATOR (PAPI)
- ROADWAY
- RUNWAY END IDENTIFIER LIGHTS (REIL)
- RUNWAY OBJECT FREE AREA (OFA)
- RUNWAY OBJECT FREE ZONE (OFZ)
- RUNWAY PROTECTION ZONE (RPZ)
- RUNWAY SAFETY AREA (RSA)
- TAXIWAY OBJECT FREE AREA (TOFA)
- TO BE REMOVED
- TOPOGRAPHIC CONTOUR
- TREES
- VISUAL APPROACH SLOPE INDICATOR (VASI)
- WIND SOCK

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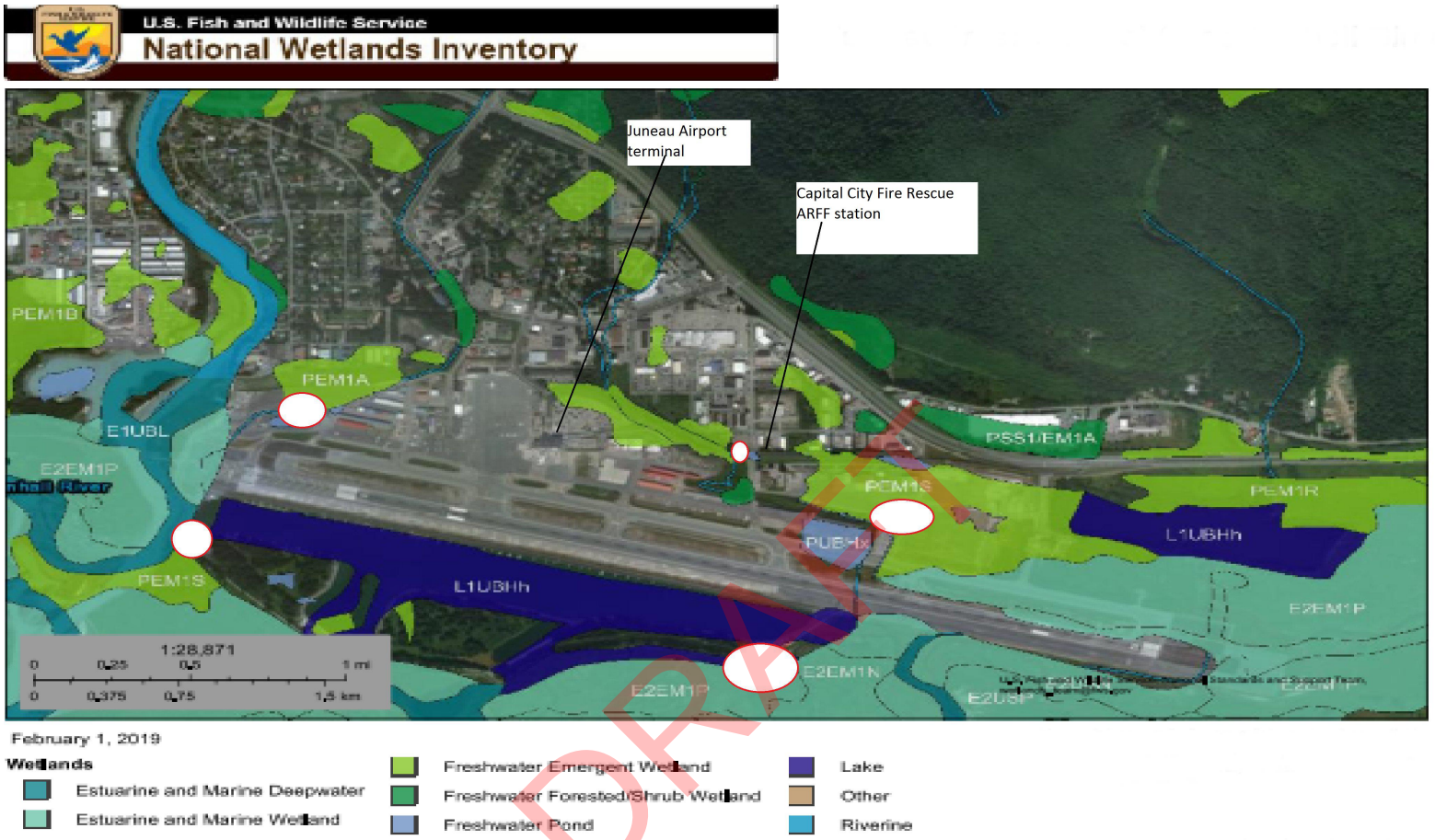
Site Plan with Soil Borings & Groundwater Monitoring Wells

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE 1"=1000'
DRAWN jmc
CHECKED jmc
DATE 2/10/2020

Fig 4

FILE: /home/dojo/Cox Environmental/ENC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 4 07 18 19.dwg
PLOTTED: 2020-02-10



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Areas outlined in red are potential places where foam was dispensed.

Image with locations provided by CCFR (2019)

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Prepared for: **City & Borough of Juneau**

FIGURE 5. POTENTIAL AFFF DISCHARGE AREAS

Juneau International Airport
1873 Shell Simmons Drive
____

Date:8.15.2019

Table 1. Summary of Soil Data – November 2019
Juneau International Airport – Juneau, AK

Analyte	CAS #	Units	Over 40-inch Human Health	Over 40-inch Migration to Groundwater	MW-1 11/16/19	MW-2 11/13/19	MW-3 11/13/19	MW-4 11/14/19	MW-5 11/18/19	MW-6 11/15/19
USEPA Method 537 (Modified)										
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	ug/Kg			ND	ND	ND	ND	ND	ND
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	ug/Kg			ND	ND	ND	ND	ND	ND
ADONA	958445-44-8	ug/Kg			ND	ND	ND	ND	ND	ND
DONA	919005-14-4	ug/Kg			ND	ND	ND	ND	ND	ND
HFPO-DA (GenX)	13252-13-6	ug/Kg			ND	ND	ND	ND	ND	ND
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	ug/Kg			ND	ND	ND	ND	ND	ND
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorobutanesulfonic acid (PFBS)	375-73-5	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorodecanoic acid (PFDA)	335-76-2	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorododecanoic acid (PFDoA)	307-55-1	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluoroheptanoic acid (PFHpA)	375-85-9	ug/Kg			ND	ND	ND	ND	ND	**0.21**
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	ug/Kg			ND	ND	ND	ND	ND	**1.2**
Perfluorohexanoic acid (PFHxA)	307-24-4	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorononanoic acid (PFNA)	375-95-1	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	ug/Kg	1300	3	**1.2**	ND	ND	ND	ND	**31 E**
Perfluorooctanoic acid (PFOA)	335-67-1	ug/Kg	1300	1.7	ND	ND	ND	ND	ND	**0.44**
Perfluorotetradecanoic acid (PFTeA)	376-06-7	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluorotridecanoic acid (PFTriA)	72629-94-8	ug/Kg			ND	ND	ND	ND	ND	ND
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ug/Kg			ND	ND	ND	ND	ND	ND

Notes:
Results in **bold/yellow** above ADEC Method 2 Soil Cleanup Level.
E - Result exceeded calibration range

DRAFT

Table 2. Summary of Groundwater Data – January 2020
Juneau International Airport – Juneau, AK

Analyte	CAS #	Units	USEPA LHA	Groundwater Cleanup Level	MW-1 01/03/20	MW-3 01/03/20	MW-4 01/03/20	MW-5 01/03/20	MW-6 01/03/20
USEPA Method 537 (Modified)									
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	ng/L			ND	ND	ND	ND	ND
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	ng/L			ND	ND	ND	ND	ND
ADONA	958445-44-8	ng/L			ND	ND	ND	ND	ND
DONA	919005-14-4	ng/L			ND	ND	ND	ND	ND
HFPO-DA (GenX)	13252-13-6	ng/L			ND	ND	ND	ND	ND
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	ng/L			ND	ND	ND	ND	ND
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	ng/L			ND	ND	ND	ND	ND
Perfluorobutanesulfonic acid (PFBS)	375-73-5	ng/L			**27**	ND	**670 E**	ND	**16**
Perfluorodecanoic acid (PFDA)	335-76-2	ng/L			ND	**2.4**	ND	ND	ND
Perfluorododecanoic acid (PFDoA)	307-55-1	ng/L			ND	ND	ND	ND	ND
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/L			**37**	**24**	**390 E**	ND	**41**
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	ng/L			**220**	**28**	**1900 E**	**1.8**	**110**
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/L			**130**	**35**	**1500 E**	ND	**73**
Perfluorononanoic acid (PFNA)	375-95-1	ng/L			**3.1**	**10**	**5.0**	ND	**6.5**
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	ng/L		400	**340 E**	**750 E**	**67**	**3.4**	**540 E**
Perfluorooctanoic acid (PFOA)	335-67-1	ng/L		400	**29**	**34**	**410 E**	ND	**40**
Perfluorotetradecanoic acid (PFTeA)	376-06-7	ng/L			ND	ND	ND	ND	ND
Perfluorotridecanoic acid (PFTriA)	72629-94-8	ng/L			ND	ND	ND	ND	ND
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/L			ND	ND	ND	ND	ND
PFOS + PFOA (calculated)		ng/L	70		**369**	**784**	**477**	**3.4**	**580**

Notes:
Results in **bold/yellow** above ADEC Method 2 Soil Cleanup Level.
E - Result exceeded calibration range

DRAFT

ATTACHMENT B

DRAFT

eurofins | Environment Testing TestAmerica

ANALYTICAL REPORT

Eurofins TestAmerica, Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Tel: (916)373-5600

Laboratory Job ID: 320-56434-1
Client Project/Site: PFAS Compounds

For:
Cox Environmental Services
712 W 12th Street
Juneau, Alaska 99801

Attn: Jolene Cox

DRAFT

Authorized for release by:
12/12/2019 4:43:40 PM
Sheri Cruz, Project Manager I
(253)922-2310
sheri.cruz@testamericainc.com

LINKS

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Have a Question? Ask The Expert

Visit us at: www.testamericainc.com

The test results in this report meet all 2003 NELAC and 2009 TNI requirements for accredited parameters, exceptions are noted in this report. This report may not be reproduced except in full, and with written approval from the laboratory. For questions please contact the Project Manager at the e-mail address or telephone number listed on this page.

This report has been electronically signed and authorized by the signatory. Electronic signature is intended to be the legally binding equivalent of a traditionally handwritten signature.

Results relate only to the items tested and the sample(s) as received by the laboratory.

Table of Contents

DRAFT

Definitions/Glossary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Qualifiers

LCMS

Qualifier	Qualifier Description
E	Result exceeded calibration range.

General Chemistry

Qualifier	Qualifier Description
F3	Duplicate RPD exceeds the control limit

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
PQL	Practical Quantitation Limit
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)

DRAFT

Job ID: 320-56434-1

Laboratory: Eurofins TestAmerica, Sacramento

Narrative

Job Narrative
320-56434-1

Comments
No additional comments.

Receipt
The samples were received on 11/20/2019 10:10 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 6.1° C.

Receipt Exceptions
The following samples were received at the laboratory outside the required temperature criteria: JIA-EB-1 (320-56434-1), JIA-MW-2 (320-56434-2), JIA-MW-3 (320-56434-3), JIA-IR-1 (320-56434-4), JIA-MW-4 (320-56434-5), JIA-MW-6 (320-56434-6), JIA-MW-6-1 (320-56434-7), JIA-EB-2 (320-56434-8), JIA-MW-1 (320-56434-9) and JIA-MW-5 (320-56434-10). Cooler received out of temp. Temp of 6.1C. Two ziplock bags of ice in cooler with samples and empty containers. Not enough ice.

The container label for the following sample did not match the information listed on the Chain-of-Custody (COC): JIA-MW-4 (320-56434-5). COC lists matrix as W (Water), but sample received was a solid/soil sample. Logged in as soil sample.

LCMS
Method 537 (modified): Due to a shortage in the marketplace for 13C3-PFBS, the target analyte PFBS and/or Perfluoropentanesulfonic acid (PFPeS) could not be quantitated against 13C3-PFBS (its labeled variant) as listed in the SOP. PFBS and Perfluoropentanesulfonic acid (PFPeS) was quantitated versus 18O2-PFHxS instead. (ICV 320-343569/9)

Method 537 (modified): The concentration of Perfluorooctanesulfonic acid (PFOS) associated with the following samples exceeded the instrument calibration range: JIA-MW-6 (320-56434-6) and JIA-MW-6-1 (320-56434-7). These analytes have been qualified; however, the peaks did not saturate the instrument detector. Historical data indicate that for the isotope dilution method, dilution and re-analysis will not produce significantly different results from those reported above the calibration range.

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

General Chemistry
Method Moisture: The sample duplicate (DUP) precision for analytical batch 320-340594 was outside control limits. Sample non-homogeneity is suspected; matrix had pebbles and sand. Data is being reported with this narration. JIA-MW-5 (320-56434-10) and (320-56434-B-10 DU)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Organic Prep
Method 3535: Insufficient sample volume was available to perform a matrix spike/matrix spike duplicate (MS/MSD) associated with preparation batch 320-341271

Method SHAKE: The following samples: JIA-MW-2 (320-56434-2) were yellow after the final volume.

PFC_IDA
Solid
320-341898

Method SHAKE: Due to loss of MB in original batch, the following samples were re-prepped. The MB fell during the transfer step and spilled: JIA-MW-2 (320-56434-2), JIA-MW-3 (320-56434-3), JIA-MW-4 (320-56434-5), JIA-MW-6 (320-56434-6), JIA-MW-6-1 (320-56434-7), JIA-MW-1 (320-56434-9) and JIA-MW-5 (320-56434-10).

Method: PFC_IDA
Matrix: Solid
Prep Batch: 320-341898

DRAFT

Job ID: 320-56434-1 (Continued)

Laboratory: Eurofins TestAmerica, Sacramento (Continued)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

DRAFT

Detection Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Client Sample ID: JIA-EB-1 — Lab Sample ID: 320-56434-1

No Detections.

Client Sample ID: JIA-MW-2 — Lab Sample ID: 320-56434-2

No Detections.

Client Sample ID: JIA-MW-3 — Lab Sample ID: 320-56434-3

No Detections.

Client Sample ID: JIA-IR-1 — Lab Sample ID: 320-56434-4

No Detections.

Client Sample ID: JIA-MW-4 — Lab Sample ID: 320-56434-5

No Detections.

Client Sample ID: JIA-MW-6 — Lab Sample ID: 320-56434-6

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanesulfonic acid (PFHxS)	1.2		0.21		ug/Kg	1	☼	537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	0.21		0.21		ug/Kg	1	☼	537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	0.44		0.21		ug/Kg	1	☼	537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	27	E	0.52		ug/Kg	1	☼	537 (modified)	Total/NA

Client Sample ID: JIA-MW-6-1 — Lab Sample ID: 320-56434-7

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanesulfonic acid (PFHxS)	0.87		0.21		ug/Kg	1	☼	537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	0.39		0.21		ug/Kg	1	☼	537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	31	E	0.54		ug/Kg	1	☼	537 (modified)	Total/NA

Client Sample ID: JIA-EB-2 — Lab Sample ID: 320-56434-8

No Detections.

Client Sample ID: JIA-MW-1 — Lab Sample ID: 320-56434-9

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorooctanesulfonic acid (PFOS)	1.2		0.54		ug/Kg	1	☼	537 (modified)	Total/NA

Client Sample ID: JIA-MW-5 — Lab Sample ID: 320-56434-10

No Detections.

DRAFT

This Detection Summary does not include radiochemical test results.

Client Sample ID: JIA-EB-1

Lab Sample ID: 320-56434-1
Matrix: Water
Date Collected: 11/13/19 11:15
Date Received: 11/20/19 10:10

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1
Perfluorohexanesulfonic acid (PFHxS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1
Perfluoroheptanoic acid (PFHpA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1
Perfluorooctanoic acid (PFOA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1
Perfluorooctanesulfonic acid (PFOS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1
Perfluorononanoic acid (PFNA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 13:58	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*109*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:58*	*1*
13C4 PFHpA	*103*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:58*	*1*
13C4 PFOA	*102*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:58*	*1*
13C4 PFOS	*102*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:58*	*1*
13C5 PFNA	*105*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:58*	*1*

Client Sample ID: JIA-MW-2

Lab Sample ID: 320-56434-2
Matrix: Solid
Date Collected: 11/13/19 12:16
Date Received: 11/20/19 10:10
Percent Solids: 69.3

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.28		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1
Perfluorohexanesulfonic acid (PFHxS)	ND		0.28		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1
Perfluoroheptanoic acid (PFHpA)	ND		0.28		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1
Perfluorooctanoic acid (PFOA)	ND		0.28		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1
Perfluorooctanesulfonic acid (PFOS)	ND		0.71		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1
Perfluorononanoic acid (PFNA)	ND		0.28		ug/Kg	☼	11/27/19 08:14	12/11/19 10:18	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*86*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:18*	*1*
13C4 PFHpA	*88*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:18*	*1*
13C4 PFOA	*83*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:18*	*1*
13C4 PFOS	*81*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:18*	*1*
13C5 PFNA	*83*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:18*	*1*

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	**30.7**		0.1		%			11/21/19 15:53	1
Percent Solids	**69.3**		0.1		%			11/21/19 15:53	1

Client Sample ID: JIA-MW-3

Lab Sample ID: 320-56434-3
Matrix: Solid
Date Collected: 11/13/19 16:14
Date Received: 11/20/19 10:10
Percent Solids: 83.9

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1
Perfluorohexanesulfonic acid (PFHxS)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1
Perfluoroheptanoic acid (PFHpA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1
Perfluorooctanoic acid (PFOA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1
Perfluorooctanesulfonic acid (PFOS)	ND		0.55		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1
Perfluorononanoic acid (PFNA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 10:28	1

DRAFT

Client Sample ID: JIA-MW-3

Lab Sample ID: 320-56434-3
Date Collected: 11/13/19 16:14
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 83.9

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*91*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:28*	*1*
13C4 PFHpA	*90*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:28*	*1*
13C4 PFOA	*83*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:28*	*1*
13C4 PFOS	*83*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:28*	*1*
13C5 PFNA	*87*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:28*	*1*

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	**16.1**		0.1		%			11/21/19 15:53	1
Percent Solids	**83.9**		0.1		%			11/21/19 15:53	1

Client Sample ID: JIA-IR-1

Lab Sample ID: 320-56434-4
Date Collected: 11/14/19 11:10
Matrix: Water
Date Received: 11/20/19 10:10

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1
Perfluorohexanesulfonic acid (PFHxS)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1
Perfluoroheptanoic acid (PFHpA)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1
Perfluorooctanoic acid (PFOA)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1
Perfluorooctanesulfonic acid (PFOS)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1
Perfluorononanoic acid (PFNA)	ND		1.9		ng/L		11/25/19 06:27	11/26/19 14:08	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*106*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:08*	*1*
13C4 PFHpA	*106*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:08*	*1*
13C4 PFOA	*104*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:08*	*1*
13C4 PFOS	*97*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:08*	*1*
13C5 PFNA	*103*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:08*	*1*

Client Sample ID: JIA-MW-4

Lab Sample ID: 320-56434-5
Date Collected: 11/14/19 12:15
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 90.5

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.20		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1
Perfluorohexanesulfonic acid (PFHxS)	ND		0.20		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1
Perfluoroheptanoic acid (PFHpA)	ND		0.20		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1
Perfluorooctanoic acid (PFOA)	ND		0.20		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1
Perfluorooctanesulfonic acid (PFOS)	ND		0.51		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1
Perfluorononanoic acid (PFNA)	ND		0.20		ug/Kg	☼	11/27/19 08:14	12/11/19 10:38	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*98*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:38*	*1*
13C4 PFHpA	*98*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:38*	*1*
13C4 PFOA	*87*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:38*	*1*
13C4 PFOS	*89*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:38*	*1*
13C5 PFNA	*88*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:38*	*1*

DRAFT

Client Sample ID: JIA-MW-4

Lab Sample ID: 320-56434-5
Date Collected: 11/14/19 12:15
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 90.5

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	**9.5**		0.1		%			11/21/19 15:53	1
Percent Solids	**90.5**		0.1		%			11/21/19 15:53	1

Client Sample ID: JIA-MW-6

Lab Sample ID: 320-56434-6
Date Collected: 11/15/19 12:17
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 89.2

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1
Perfluorohexanesulfonic acid (PFHxS)	**1.2**		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1
Perfluoroheptanoic acid (PFHpA)	**0.21**		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1
Perfluorooctanoic acid (PFOA)	**0.44**		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1
Perfluorooctanesulfonic acid (PFOS)	**27**	**E**	0.52		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1
Perfluorononanoic acid (PFNA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:48	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*91*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:48*	*1*
13C4 PFHpA	*90*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:48*	*1*
13C4 PFOA	*84*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:48*	*1*
13C4 PFOS	*83*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:48*	*1*
13C5 PFNA	*79*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:48*	*1*

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	**10.8**		0.1		%			11/21/19 15:53	1
Percent Solids	**89.2**		0.1		%			11/21/19 15:53	1

Client Sample ID: JIA-MW-6-1

Lab Sample ID: 320-56434-7
Date Collected: 11/15/19 12:19
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 90.2

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1
Perfluorohexanesulfonic acid (PFHxS)	**0.87**		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1
Perfluoroheptanoic acid (PFHpA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1
Perfluorooctanoic acid (PFOA)	**0.39**		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1
Perfluorooctanesulfonic acid (PFOS)	**31**	**E**	0.54		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1
Perfluorononanoic acid (PFNA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 10:58	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*86*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:58*	*1*
13C4 PFHpA	*88*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:58*	*1*
13C4 PFOA	*84*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:58*	*1*
13C4 PFOS	*81*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:58*	*1*
13C5 PFNA	*79*		*25 - 150*	*11/27/19 08:14*	*12/11/19 10:58*	*1*

DRAFT

Client Sample ID: JIA-MW-6-1

Lab Sample ID: 320-56434-7
Date Collected: 11/15/19 12:19
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 90.2

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	9.8		0.1		%			11/21/19 15:53	1
Percent Solids	90.2		0.1		%			11/21/19 15:53	1

Client Sample ID: JIA-EB-2

Lab Sample ID: 320-56434-8
Date Collected: 11/16/19 10:00
Matrix: Water
Date Received: 11/20/19 10:10

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1
Perfluorohexanesulfonic acid (PFHxS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1
Perfluoroheptanoic acid (PFHpA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1
Perfluorooctanoic acid (PFOA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1
Perfluorooctanesulfonic acid (PFOS)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1
Perfluorononanoic acid (PFNA)	ND		1.8		ng/L		11/25/19 06:27	11/26/19 14:18	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*108*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:18*	*1*
13C4 PFHpA	*104*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:18*	*1*
13C4 PFOA	*100*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:18*	*1*
13C4 PFOS	*101*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:18*	*1*
13C5 PFNA	*105*		*25 - 150*	*11/25/19 06:27*	*11/26/19 14:18*	*1*

Client Sample ID: JIA-MW-1

Lab Sample ID: 320-56434-9
Date Collected: 11/16/19 13:52
Matrix: Solid
Date Received: 11/20/19 10:10
Percent Solids: 87.4

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1
Perfluorohexanesulfonic acid (PFHxS)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1
Perfluoroheptanoic acid (PFHpA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1
Perfluorooctanoic acid (PFOA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1
Perfluorooctanesulfonic acid (PFOS)	1.2		0.54		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1
Perfluorononanoic acid (PFNA)	ND		0.22		ug/Kg	☼	11/27/19 08:14	12/11/19 11:08	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*81*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:08*	*1*
13C4 PFHpA	*80*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:08*	*1*
13C4 PFOA	*78*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:08*	*1*
13C4 PFOS	*78*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:08*	*1*
13C5 PFNA	*78*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:08*	*1*

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	12.6		0.1		%			11/21/19 15:53	1
Percent Solids	87.4		0.1		%			11/21/19 15:53	1

DRAFT

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Client Sample ID: JIA-MW-5

Date Collected: 11/18/19 10:26
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-10

Matrix: Solid
Percent Solids: 89.0

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1
Perfluorohexanesulfonic acid (PFHxS)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1
Perfluoroheptanoic acid (PFHpA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1
Perfluorooctanoic acid (PFOA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1
Perfluorooctanesulfonic acid (PFOS)	ND		0.53		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1
Perfluorononanoic acid (PFNA)	ND		0.21		ug/Kg	☼	11/27/19 08:14	12/11/19 11:18	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*93*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:18*	*1*
13C4 PFHpA	*93*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:18*	*1*
13C4 PFOA	*86*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:18*	*1*
13C4 PFOS	*82*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:18*	*1*
13C5 PFNA	*84*		*25 - 150*	*11/27/19 08:14*	*12/11/19 11:18*	*1*

General Chemistry

Analyte	Result	Qualifier	RL	RL	Unit	D	Prepared	Analyzed	Dil Fac
Percent Moisture	**11.0**		0.1		%			11/21/19 15:53	1
Percent Solids	**89.0**		0.1		%			11/21/19 15:53	1

DRAFT

Isotope Dilution Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Method: 537 (modified) - Fluorinated Alkyl Substances

Matrix: Solid

Prep Type: Total/NA

		Percent Isotope Dilution Recovery (Acceptance Limits)				
Lab Sample ID	Client Sample ID	PFHxS (25-150)	PFHpA (25-150)	PFOA (25-150)	PFOS (25-150)	PFNA (25-150)
320-56434-2	JIA-MW-2	86	88	83	81	83
320-56434-3	JIA-MW-3	91	90	83	83	87
320-56434-5	JIA-MW-4	98	98	87	89	88
320-56434-6	JIA-MW-6	91	90	84	83	79
320-56434-7	JIA-MW-6-1	86	88	84	81	79
320-56434-9	JIA-MW-1	81	80	78	78	78
320-56434-10	JIA-MW-5	93	93	86	82	84

Surrogate Legend

PFHxS = 18O2 PFHxS
PFHpA = 13C4 PFHpA
PFOA = 13C4 PFOA
PFOS = 13C4 PFOS
PFNA = 13C5 PFNA

Method: 537 (modified) - Fluorinated Alkyl Substances

Matrix: Water

Prep Type: Total/NA

		Percent Isotope Dilution Recovery (Acceptance Limits)				
Lab Sample ID	Client Sample ID	PFHxS (25-150)	PFHpA (25-150)	PFOA (25-150)	PFOS (25-150)	PFNA (25-150)
320-56434-1	JIA-EB-1	109	103	102	102	105
320-56434-4	JIA-IR-1	106	106	104	97	103
320-56434-8	JIA-EB-2	108	104	100	101	105
LCS 320-341271/2-A	Lab Control Sample	110	107	103	107	107
LCSD 320-341271/3-A	Lab Control Sample Dup	107	103	103	97	102
MB 320-341271/1-A	Method Blank	109	107	106	99	106

Surrogate Legend

PFHxS = 18O2 PFHxS
PFHpA = 13C4 PFHpA
PFOA = 13C4 PFOA
PFOS = 13C4 PFOS
PFNA = 13C5 PFNA

DRAFT

Method: 537 (modified) - Fluorinated Alkyl Substances

Lab Sample ID: MB 320-341271/1-A
Matrix: Water
Analysis Batch: 341716

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 341271

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanesulfonic acid (PFBS)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1
Perfluorohexanesulfonic acid (PFHxS)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1
Perfluoroheptanoic acid (PFHpA)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1
Perfluorooctanoic acid (PFOA)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1
Perfluorooctanesulfonic acid (PFOS)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1
Perfluorononanoic acid (PFNA)	ND		2.0		ng/L		11/25/19 06:27	11/26/19 13:28	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
18O2 PFHxS	*109*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:28*	*1*
13C4 PFHpA	*107*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:28*	*1*
13C4 PFOA	*106*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:28*	*1*
13C4 PFOS	*99*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:28*	*1*
13C5 PFNA	*106*		*25 - 150*	*11/25/19 06:27*	*11/26/19 13:28*	*1*

Lab Sample ID: LCS 320-341271/2-A
Matrix: Water
Analysis Batch: 341716

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 341271

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Perfluorobutanesulfonic acid (PFBS)	35.4	32.6		ng/L		92	67 - 127
Perfluorohexanesulfonic acid (PFHxS)	36.4	33.3		ng/L		91	59 - 119
Perfluoroheptanoic acid (PFHpA)	40.0	38.4		ng/L		96	72 - 132
Perfluorooctanoic acid (PFOA)	40.0	39.3		ng/L		98	70 - 130
Perfluorooctanesulfonic acid (PFOS)	37.1	32.0		ng/L		86	70 - 130
Perfluorononanoic acid (PFNA)	40.0	40.0		ng/L		100	75 - 135

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
18O2 PFHxS	*110*		*25 - 150*
13C4 PFHpA	*107*		*25 - 150*
13C4 PFOA	*103*		*25 - 150*
13C4 PFOS	*107*		*25 - 150*
13C5 PFNA	*107*		*25 - 150*

Lab Sample ID: LCSD 320-341271/3-A
Matrix: Water
Analysis Batch: 341716

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 341271

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Perfluorobutanesulfonic acid (PFBS)	35.4	32.7		ng/L		92	67 - 127	0	30
Perfluorohexanesulfonic acid (PFHxS)	36.4	32.6		ng/L		90	59 - 119	2	30
Perfluoroheptanoic acid (PFHpA)	40.0	38.2		ng/L		95	72 - 132	1	30
Perfluorooctanoic acid (PFOA)	40.0	39.2		ng/L		98	70 - 130	0	30
Perfluorooctanesulfonic acid (PFOS)	37.1	34.9		ng/L		94	70 - 130	9	30
Perfluorononanoic acid (PFNA)	40.0	40.8		ng/L		102	75 - 135	2	30

DRAFT

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Isotope Dilution	LCSD %Recovery	LCSD Qualifier	Limits
18O2 PFHxS	*107*		*25 - 150*
13C4 PFHpA	*103*		*25 - 150*
13C4 PFOA	*103*		*25 - 150*
13C4 PFOS	*97*		*25 - 150*
13C5 PFNA	*102*		*25 - 150*

Method: D 2216 - Percent Moisture

Lab Sample ID: 320-56434-10 DU
Matrix: Solid
Analysis Batch: 340594

Client Sample ID: JIA-MW-5
Prep Type: Total/NA

Analyte	Sample Result	Sample Qualifier		DU Result	DU Qualifier	Unit	D			RPD	RPD Limit
Percent Moisture	11.0			13.8	F3	%				22	20
Percent Solids	89.0			86.2		%				3	20

DRAFT

LCMS

Prep Batch: 341271

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-56434-1	JIA-EB-1	Total/NA	Water	3535	
320-56434-4	JIA-IR-1	Total/NA	Water	3535	
320-56434-8	JIA-EB-2	Total/NA	Water	3535	
MB 320-341271/1-A	Method Blank	Total/NA	Water	3535	
LCS 320-341271/2-A	Lab Control Sample	Total/NA	Water	3535	
LCSD 320-341271/3-A	Lab Control Sample Dup	Total/NA	Water	3535	

Analysis Batch: 341716

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-56434-1	JIA-EB-1	Total/NA	Water	537 (modified)	341271
320-56434-4	JIA-IR-1	Total/NA	Water	537 (modified)	341271
320-56434-8	JIA-EB-2	Total/NA	Water	537 (modified)	341271
MB 320-341271/1-A	Method Blank	Total/NA	Water	537 (modified)	341271
LCS 320-341271/2-A	Lab Control Sample	Total/NA	Water	537 (modified)	341271
LCSD 320-341271/3-A	Lab Control Sample Dup	Total/NA	Water	537 (modified)	341271

Prep Batch: 341898

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-56434-2	JIA-MW-2	Total/NA	Solid	SHAKE	
320-56434-3	JIA-MW-3	Total/NA	Solid	SHAKE	
320-56434-5	JIA-MW-4	Total/NA	Solid	SHAKE	
320-56434-6	JIA-MW-6	Total/NA	Solid	SHAKE	
320-56434-7	JIA-MW-6-1	Total/NA	Solid	SHAKE	
320-56434-9	JIA-MW-1	Total/NA	Solid	SHAKE	
320-56434-10	JIA-MW-5	Total/NA	Solid	SHAKE	

Analysis Batch: 344571

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-56434-2	JIA-MW-2	Total/NA	Solid	537 (modified)	341898
320-56434-3	JIA-MW-3	Total/NA	Solid	537 (modified)	341898
320-56434-5	JIA-MW-4	Total/NA	Solid	537 (modified)	341898
320-56434-6	JIA-MW-6	Total/NA	Solid	537 (modified)	341898
320-56434-7	JIA-MW-6-1	Total/NA	Solid	537 (modified)	341898
320-56434-9	JIA-MW-1	Total/NA	Solid	537 (modified)	341898
320-56434-10	JIA-MW-5	Total/NA	Solid	537 (modified)	341898

General Chemistry

Analysis Batch: 340594

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-56434-2	JIA-MW-2	Total/NA	Solid	D 2216	
320-56434-3	JIA-MW-3	Total/NA	Solid	D 2216	
320-56434-5	JIA-MW-4	Total/NA	Solid	D 2216	
320-56434-6	JIA-MW-6	Total/NA	Solid	D 2216	
320-56434-7	JIA-MW-6-1	Total/NA	Solid	D 2216	
320-56434-9	JIA-MW-1	Total/NA	Solid	D 2216	
320-56434-10	JIA-MW-5	Total/NA	Solid	D 2216	
320-56434-10 DU	JIA-MW-5	Total/NA	Solid	D 2216	

DRAFT

Lab Chronicle

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Client Sample ID: JIA-EB-1
Date Collected: 11/13/19 11:15
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-1
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			278.1 mL	10.0 mL	341271	11/25/19 06:27	MTN	TAL SAC
Total/NA	Analysis	537 (modified)		1			341716	11/26/19 13:58	S1M	TAL SAC

Client Sample ID: JIA-MW-2
Date Collected: 11/13/19 12:16
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-2
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-2
Date Collected: 11/13/19 12:16
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-2
Matrix: Solid
Percent Solids: 69.3

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.07 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 10:18	S1M	TAL SAC

Client Sample ID: JIA-MW-3
Date Collected: 11/13/19 16:14
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-3
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-3
Date Collected: 11/13/19 16:14
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-3
Matrix: Solid
Percent Solids: 83.9

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.41 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 10:28	S1M	TAL SAC

Client Sample ID: JIA-IR-1
Date Collected: 11/14/19 11:10
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-4
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			268.2 mL	10.0 mL	341271	11/25/19 06:27	MTN	TAL SAC
Total/NA	Analysis	537 (modified)		1			341716	11/26/19 14:08	S1M	TAL SAC

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Lab Chronicle

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Client Sample ID: JIA-MW-4
Date Collected: 11/14/19 12:15
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-5
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-4
Date Collected: 11/14/19 12:15
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-5
Matrix: Solid
Percent Solids: 90.5

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.46 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 10:38	S1M	TAL SAC

Client Sample ID: JIA-MW-6
Date Collected: 11/15/19 12:17
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-6
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-6
Date Collected: 11/15/19 12:17
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-6
Matrix: Solid
Percent Solids: 89.2

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.41 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 10:48	S1M	TAL SAC

Client Sample ID: JIA-MW-6-1
Date Collected: 11/15/19 12:19
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-7
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-6-1
Date Collected: 11/15/19 12:19
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-7
Matrix: Solid
Percent Solids: 90.2

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.16 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 10:58	S1M	TAL SAC

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Lab Chronicle

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Client Sample ID: JIA-EB-2
Date Collected: 11/16/19 10:00
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-8
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			274.2 mL	10.0 mL	341271	11/25/19 06:27	MTN	TAL SAC
Total/NA	Analysis	537 (modified)		1			341716	11/26/19 14:18	S1M	TAL SAC

Client Sample ID: JIA-MW-1
Date Collected: 11/16/19 13:52
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-9
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-1
Date Collected: 11/16/19 13:52
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-9
Matrix: Solid
Percent Solids: 87.4

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.28 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 11:08	S1M	TAL SAC

Client Sample ID: JIA-MW-5
Date Collected: 11/18/19 10:26
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-10
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	D 2216		1			340594	11/21/19 15:53	HRB	TAL SAC

Client Sample ID: JIA-MW-5
Date Collected: 11/18/19 10:26
Date Received: 11/20/19 10:10

Lab Sample ID: 320-56434-10
Matrix: Solid
Percent Solids: 89.0

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	SHAKE			5.33 g	10.00 mL	341898	11/27/19 08:14	AEC	TAL SAC
Total/NA	Analysis	537 (modified)		1			344571	12/11/19 11:18	S1M	TAL SAC

Laboratory References:

TAL SAC = Eurofins TestAmerica, Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

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Accreditation/Certification Summary

Client: Cox Environmental Services
Job ID: 320-56434-1
Project/Site: PFAS Compounds

Laboratory: Eurofins TestAmerica, Sacramento

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State	17-020	01-20-21
ANAB	Dept. of Defense ELAP	L2468	01-20-21
ANAB	Dept. of Energy	L2468.01	01-20-21
ANAB	ISO/IEC 17025	L2468	01-20-21
Arizona	State	AZ0708	08-11-20
Arkansas DEQ	State	19-042-0	06-17-20
California	State	2897	01-31-20
Colorado	State	CA0004	08-31-20
Connecticut	State	PH-0691	06-30-21
Florida	NELAP	E87570	06-30-20
Georgia	State	4040	01-29-20
Hawaii	State	<cert No.>	01-29-20
Illinois	NELAP	200060	03-17-20
Kansas	NELAP	E-10375	10-31-20 *
Louisiana	NELAP	01944	06-30-20
Maine	State	2018009	04-14-20
Michigan	State	9947	01-29-20
Michigan	State Program	9947	01-31-20
Nevada	State	CA000442020-1	07-31-20
New Hampshire	NELAP	2997	04-18-20
New Jersey	NELAP	CA005	06-30-20
New York	NELAP	11666	04-01-20
Oregon	NELAP	4040	01-29-20
Pennsylvania	NELAP	68-01272	03-31-20
Texas	NELAP	T104704399-19-13	05-31-20
US Fish & Wildlife	US Federal Programs	58448	07-31-20
USDA	US Federal Programs	P330-18-00239	07-31-21
Utah	NELAP	CA000442019-01	02-29-20
Vermont	State	VT-4040	04-16-20
Virginia	NELAP	460278	03-14-20
Washington	State	C581	05-05-20
West Virginia (DW)	State	9930C	12-31-19
Wyoming	State Program	8TMS-L	01-28-19 *

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Laboratory: Eurofins TestAmerica, Seattle

The accreditations/certifications listed below are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State Program	17-024	01-19-20

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

Method Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Method	Method Description	Protocol	Laboratory
537 (modified)	Fluorinated Alkyl Substances	EPA	TAL SAC
D 2216	Percent Moisture	ASTM	TAL SAC
3535	Solid-Phase Extraction (SPE)	SW846	TAL SAC
SHAKE	Shake Extraction with Ultrasonic Bath Extraction	SW846	TAL SAC

Protocol References:

ASTM = ASTM International

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

Laboratory References:

TAL SAC = Eurofins TestAmerica, Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

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Sample Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-56434-1

Lab Sample ID	Client Sample ID	Matrix	Collected	Received	Asset ID
320-56434-1	JIA-EB-1	Water	11/13/19 11:15	11/20/19 10:10	
320-56434-2	JIA-MW-2	Solid	11/13/19 12:16	11/20/19 10:10	
320-56434-3	JIA-MW-3	Solid	11/13/19 16:14	11/20/19 10:10	
320-56434-4	JIA-IR-1	Water	11/14/19 11:10	11/20/19 10:10	
320-56434-5	JIA-MW-4	Solid	11/14/19 12:15	11/20/19 10:10	
320-56434-6	JIA-MW-6	Solid	11/15/19 12:17	11/20/19 10:10	
320-56434-7	JIA-MW-6-1	Solid	11/15/19 12:19	11/20/19 10:10	
320-56434-8	JIA-EB-2	Water	11/16/19 10:00	11/20/19 10:10	
320-56434-9	JIA-MW-1	Solid	11/16/19 13:52	11/20/19 10:10	
320-56434-10	JIA-MW-5	Solid	11/18/19 10:26	11/20/19 10:10	

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Chain of Custody Record

327850

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Laboratories, Inc.

TAL-8210 (0713)

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

Client Contact	Project Manager: J. COX	Site Contact: S. CRUZ	Date: 11.13.19	COC No: JIA-1-A
Company Name: COX ENVIRONMENTAL	Tel/Fax: 907.586.4447	Lab Contact: J. COX	Carrier: AK-AIR	1 of ____ COCs
Address: 712 W 12TH ST	Analysis Turnaround Time			Sampler: J. COX
City/State/Zip: JUNEAU AK 99801	☐ CALENDAR DAYS ☒ WORKING DAYS			For Lab Use Only:
Phone: 907.586.4447	TAT if different from Below ____			Walk-in Client:
Fax:	☒ 2 weeks			Lab Sampling:
Project Name: JIA PFAS	☐ 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)	537 PFAS	% MOISTURE	Sample Specific Notes:
JIA-EB-1	11.13.19	11:15	G	W	2			X		
JIA-MW-2	11.13.19	12:16	G	S	2			X	X	
JIA-MW-3	11.13.19	16:14	G	S	2			X	X	
JIA-IR-1	11.14.19	11:10	G	W	2			X		
JIA-MW-4	11.14.19	12:15	G	W	2			X	X	
JIA-MW-6	11.15.19	12:17	G	S	2			X	X	
JIA-MW-6-1	11.15.19	12:19	G	S	2			X	X	
JIA-EB-2	11.16.19	10:00	G	W	2			X		
JIA-MW-1	11.16.19	13:52	G	S	2			X	X	
JIA-MW-5	11.18.19	10:26	G	S	2			X	X	

320-58434 Chain of Custody

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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: 6.3°C Corr'd: 6.1°C | Therm ID No.: AK-5

Relinquished by:	Company:	Date/Time: 11.19.19 13:30	Received by:	Company: EA WSC	Date/Time: 20 Nov 19 10:10
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

* Sample 2/2 are soil COC Matrix state water JG 11/20/19

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12/12/2019

Login Sample Receipt Checklist

Client: Cox Environmental Services

Job Number: 320-56434-1

Login Number: 56434
List Number: 1
Creator: Her, David A

List Source: Eurofins TestAmerica, Sacramento

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	188109/188108
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	False	Cooler temperature outside required temperature criteria.
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	False	Refer to Job Narrative for details.
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

DRAFT

eurofins | Environment Testing TestAmerica

ANALYTICAL REPORT

Eurofins TestAmerica, Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Tel: (916)373-5600

Laboratory Job ID: 320-57452-1
Client Project/Site: PFAS Compounds

For:
Cox Environmental Services
712 W 12th Street
Juneau, Alaska 99801

Attn: Jolene Cox

Authorized for release by:
1/23/2020 4:32:11 PM
Sheri Cruz, Project Manager I
(253)922-2310
sheri.cruz@testamericainc.com

DRAFT

LINKS

Review your project results through TotalAccess

Have a Question? Ask The Expert

Visit us at: www.testamericainc.com

The test results in this report meet all 2003 NELAC and 2009 TNI requirements for accredited parameters, exceptions are noted in this report. This report may not be reproduced except in full, and with written approval from the laboratory. For questions please contact the Project Manager at the e-mail address or telephone number listed on this page.

This report has been electronically signed and authorized by the signatory. Electronic signature is intended to be the legally binding equivalent of a traditionally handwritten signature.

Results relate only to the items tested and the sample(s) as received by the laboratory.

Table of Contents

DRAFT

Definitions/Glossary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Qualifiers

LCMS

Qualifier	Qualifier Description
*	Isotope Dilution analyte is outside acceptance limits.
E	Result exceeded calibration range.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
PQL	Practical Quantitation Limit
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)

DRAFT

Job ID: 320-57452-1

Laboratory: Eurofins TestAmerica, Sacramento

Narrative

Job Narrative
320-57452-1

Comments
No additional comments.

Receipt
The samples were received on 1/7/2020 11:38 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 5.2º C.

LCMS
Method 537 (modified): 13C2 PFTeDA Isotope Dilution Analyte (IDA) recovery associated with the following samples is below the method recommended limit: MW-3 (320-57452-1). Generally, data quality is not considered affected if the IDA signal-to-noise ratio is greater than 10:1, which is achieved for all IDA in the samples.

Method 537 (modified): The concentration of Perfluorooctanoic acid (PFOA), Perfluoroheptanoic acid (PFHpA), Perfluorohexanoic acid (PFHxA), Perfluorobutanesulfonic acid (PFBS) and Perfluorohexanesulfonic acid (PFHxS) associated with the following samples exceeded the instrument calibration range: MW-4 (320-57452-2). These analytes have been qualified; however, the peaks did not saturate the instrument detector. Historical data indicate that for the isotope dilution method, dilution and re-analysis will not produce significantly different results from those reported above the calibration range.

Method 537 (modified): The concentration of Perfluorooctanesulfonic acid (PFOS) associated with the following samples exceeded the instrument calibration range: MW-1 (320-57452-4), MW-6 (320-57452-7) and MW-6-1 (320-57452-8). These analytes have been qualified; however, the peaks did not saturate the instrument detector. Historical data indicate that for the isotope dilution method, dilution and re-analysis will not produce significantly different results from those reported above the calibration range.

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Organic Prep
Method 3535: Insufficient sample volume was available to perform a matrix spike/matrix spike duplicate (MS/MSD) associated with preparation batch 320-350842. Method Code: 3535 PFC-W

Method 3535: The following samples were observed to be dark yellow prior to extraction: MW-1 (320-57452-4)

Method Code: 3535 PFC-W
preparation batch 320-350842

Method 3535: The following samples were observed to contain sediment prior to extraction: MW-3 (320-57452-1), MW-4 (320-57452-2), MW-5 (320-57452-3), MW-1 (320-57452-4), MW-6 (320-57452-7) and MW-6-1 (320-57452-8)

Method Code: 3535 PFC-W
preparation batch 320-350842

Method 3535: The following samples contain non-settleable particulate matter which clogged the solid phase extraction column: MW-3 (320-57452-1), MW-4 (320-57452-2), MW-5 (320-57452-3), MW-1 (320-57452-4), MW-6 (320-57452-7) and MW-6-1 (320-57452-8)

Method Code: 3535 PFC-W
preparation batch 320-350842

Method 3535: The following samples were observed to be yellow after final voluming: MW-3 (320-57452-1), MW-4 (320-57452-2), MW-5 (320-57452-3) and MW-1 (320-57452-4)

Method Code: 3535 PFC-W
preparation batch 320-350842

DRAFT

Job ID: 320-57452-1 (Continued)

Laboratory: Eurofins TestAmerica, Sacramento (Continued)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

DRAFT

Detection Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Client Sample ID: MW-3 — Lab Sample ID: 320-57452-1

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanoic acid (PFHxA)	35		1.9		ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	24		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	34		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	10		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorodecanoic acid (PFDA)	2.4		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	28		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	750	E	1.9		ng/L	1		537 (modified)	Total/NA

Client Sample ID: MW-4 — Lab Sample ID: 320-57452-2

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanoic acid (PFHxA)	1500	E	1.9		ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	390	E	1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	410	E	1.9		ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	5.0		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	670	E	1.9		ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1900	E	1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	67		1.9		ng/L	1		537 (modified)	Total/NA

Client Sample ID: MW-5 — Lab Sample ID: 320-57452-3

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanesulfonic acid (PFHxS)	1.8		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	3.4		1.8		ng/L	1		537 (modified)	Total/NA

Client Sample ID: MW-1 — Lab Sample ID: 320-57452-4

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanoic acid (PFHxA)	130		1.8		ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	37		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	29		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	3.1		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	27		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	220		1.8		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	340	E	1.8		ng/L	1		537 (modified)	Total/NA

Client Sample ID: IR-1 — Lab Sample ID: 320-57452-5

No Detections.

Client Sample ID: ER-1 — Lab Sample ID: 320-57452-6

No Detections.

Client Sample ID: MW-6 — Lab Sample ID: 320-57452-7

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanoic acid (PFHxA)	63		1.9		ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	34		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	36		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	5.5		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	14		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	98		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	490	E	1.9		ng/L	1		537 (modified)	Total/NA

This Detection Summary does not include radiochemical test results.

DRAFT

Detection Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Client Sample ID: MW-6-1

Lab Sample ID: 320-57452-8

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorohexanoic acid (PFHxA)	73		1.9		ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	41		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	40		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	6.5		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	16		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	110		1.9		ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	540	E	1.9		ng/L	1		537 (modified)	Total/NA

DRAFT

This Detection Summary does not include radiochemical test results.

Client Sample ID: MW-3

Date Collected: 01/03/20 10:48
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-1
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	**35**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluoroheptanoic acid (PFHpA)	**24**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorooctanoic acid (PFOA)	**34**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorononanoic acid (PFNA)	**10**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorodecanoic acid (PFDA)	**2.4**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluoroundecanoic acid (PFUnA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorododecanoic acid (PFDoA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorotridecanoic acid (PFTriA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorobutanesulfonic acid (PFBS)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorohexanesulfonic acid (PFHxS)	**28**		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
Perfluorooctanesulfonic acid (PFOS)	**750**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		19		ng/L		01/15/20 05:09	01/16/20 06:42	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		19		ng/L		01/15/20 05:09	01/16/20 06:42	1
ADONA	ND		2.0		ng/L		01/15/20 05:09	01/16/20 06:42	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
HFPO-DA (GenX)	ND		3.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1
DONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:42	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*68*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C4 PFHpA	*68*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C4 PFOA	*67*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C5 PFNA	*66*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C2 PFDA	*57*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C2 PFUnA	*59*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C2 PFDoA	*52*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C2 PFTeDA	*24*	*	*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C3 PFBS	*80*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
18O2 PFHxS	*76*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C4 PFOS	*73*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
d3-NMeFOSAA	*67*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
d5-NEtFOSAA	*70*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*
13C3 HFPO-DA	*38*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:42*	*1*

Client Sample ID: MW-4

Date Collected: 01/03/20 11:20
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-2
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	**1500**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluoroheptanoic acid (PFHpA)	**390**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorooctanoic acid (PFOA)	**410**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorononanoic acid (PFNA)	**5.0**		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1

DRAFT

Client Sample ID: MW-4

Date Collected: 01/03/20 11:20
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-2

Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorodecanoic acid (PFDA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluoroundecanoic acid (PFUnA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorododecanoic acid (PFDoA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorotridecanoic acid (PFTriA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorobutanesulfonic acid (PFBS)	**670**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorohexanesulfonic acid (PFHxS)	**1900**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
Perfluorooctanesulfonic acid (PFOS)	**67**		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		19		ng/L		01/15/20 05:09	01/16/20 06:50	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		19		ng/L		01/15/20 05:09	01/16/20 06:50	1
ADONA	ND		2.0		ng/L		01/15/20 05:09	01/16/20 06:50	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
HFPO-DA (GenX)	ND		3.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1
DONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:50	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*65*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C4 PFHpA	*64*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C4 PFOA	*67*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C5 PFNA	*81*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C2 PFDA	*82*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C2 PFUnA	*80*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C2 PFDoA	*80*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C2 PFTeDA	*54*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C3 PFBS	*87*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
18O2 PFHxS	*78*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C4 PFOS	*99*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
d3-NMeFOSAA	*95*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
d5-NEtFOSAA	*97*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*
13C3 HFPO-DA	*51*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:50*	*1*

Client Sample ID: MW-5

Date Collected: 01/03/20 11:58
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-3

Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluoroheptanoic acid (PFHpA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorooctanoic acid (PFOA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorononanoic acid (PFNA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorodecanoic acid (PFDA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluoroundecanoic acid (PFUnA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorododecanoic acid (PFDoA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1

DRAFT

Client Sample ID: MW-5

Date Collected: 01/03/20 11:58
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-3
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorotridecanoic acid (PFTriA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorobutanesulfonic acid (PFBS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorohexanesulfonic acid (PFHxS)	**1.8**		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
Perfluorooctanesulfonic acid (PFOS)	**3.4**		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		18		ng/L		01/15/20 05:09	01/16/20 06:58	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		18		ng/L		01/15/20 05:09	01/16/20 06:58	1
ADONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 06:58	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
HFPO-DA (GenX)	ND		3.6		ng/L		01/15/20 05:09	01/16/20 06:58	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1
DONA	ND		1.8		ng/L		01/15/20 05:09	01/16/20 06:58	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*63*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C4 PFHpA	*59*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C4 PFOA	*60*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C5 PFNA	*60*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C2 PFDA	*53*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C2 PFUnA	*43*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C2 PFDoA	*45*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C2 PFTeDA	*33*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C3 PFBS	*67*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
18O2 PFHxS	*65*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C4 PFOS	*62*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
d3-NMeFOSAA	*61*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
d5-NEtFOSAA	*64*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*
13C3 HFPO-DA	*56*		*25 - 150*	*01/15/20 05:09*	*01/16/20 06:58*	*1*

DRAFT

Client Sample ID: MW-1

Date Collected: 01/03/20 12:37
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-4
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	**130**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluoroheptanoic acid (PFHpA)	**37**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorooctanoic acid (PFOA)	**29**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorononanoic acid (PFNA)	**3.1**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorodecanoic acid (PFDA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluoroundecanoic acid (PFUnA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorododecanoic acid (PFDoA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorotridecanoic acid (PFTriA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorobutanesulfonic acid (PFBS)	**27**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1

Client Sample ID: MW-1

Lab Sample ID: 320-57452-4

Date Collected: 01/03/20 12:37

Matrix: Water

Date Received: 01/07/20 09:45

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanesulfonic acid (PFHxS)	**220**		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
Perfluorooctanesulfonic acid (PFOS)	**340**	**E**	1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		18		ng/L		01/15/20 05:09	01/16/20 07:06	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		18		ng/L		01/15/20 05:09	01/16/20 07:06	1
ADONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:06	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
HFPO-DA (GenX)	ND		3.6		ng/L		01/15/20 05:09	01/16/20 07:06	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1
DONA	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:06	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*74*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C4 PFHpA	*73*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C4 PFOA	*74*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C5 PFNA	*69*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C2 PFDA	*60*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C2 PFUnA	*64*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C2 PFDoA	*61*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C2 PFTeDA	*44*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C3 PFBS	*82*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
18O2 PFHxS	*78*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C4 PFOS	*76*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
d3-NMeFOSAA	*74*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
d5-NEtFOSAA	*77*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*
13C3 HFPO-DA	*57*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:06*	*1*

DRAFT

Client Sample ID: IR-1

Lab Sample ID: 320-57452-5

Date Collected: 01/03/20 13:30

Matrix: Water

Date Received: 01/07/20 09:45

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluoroheptanoic acid (PFHpA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorooctanoic acid (PFOA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorononanoic acid (PFNA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorodecanoic acid (PFDA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluoroundecanoic acid (PFUnA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorododecanoic acid (PFDoA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorotridecanoic acid (PFTriA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorobutanesulfonic acid (PFBS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorohexanesulfonic acid (PFHxS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
Perfluorooctanesulfonic acid (PFOS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		18		ng/L		01/15/20 05:09	01/16/20 07:14	1

Client Sample ID: IR-1

Date Collected: 01/03/20 13:30
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-5
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		18		ng/L		01/15/20 05:09	01/16/20 07:14	1
ADONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:14	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
HFPO-DA (GenX)	ND		3.6		ng/L		01/15/20 05:09	01/16/20 07:14	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1
DONA	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:14	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*105*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C4 PFHpA	*103*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C4 PFOA	*96*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C5 PFNA	*95*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C2 PFDA	*95*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C2 PFUnA	*87*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C2 PFDoA	*93*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C2 PFTeDA	*71*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C3 PFBS	*105*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
18O2 PFHxS	*108*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C4 PFOS	*105*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
d3-NMeFOSAA	*110*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
d5-NEtFOSAA	*112*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*
13C3 HFPO-DA	*73*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:14*	*1*

Client Sample ID: ER-1

Date Collected: 01/03/20 13:35
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-6
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluoroheptanoic acid (PFHpA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorooctanoic acid (PFOA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorononanoic acid (PFNA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorodecanoic acid (PFDA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluoroundecanoic acid (PFUnA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorododecanoic acid (PFDoA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorotridecanoic acid (PFTriA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorobutanesulfonic acid (PFBS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorohexanesulfonic acid (PFHxS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
Perfluorooctanesulfonic acid (PFOS)	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		18		ng/L		01/15/20 05:09	01/16/20 07:38	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		18		ng/L		01/15/20 05:09	01/16/20 07:38	1
ADONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:38	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
HFPO-DA (GenX)	ND		3.6		ng/L		01/15/20 05:09	01/16/20 07:38	1

DRAFT

Client Sample ID: ER-1

Lab Sample ID: 320-57452-6

Date Collected: 01/03/20 13:35

Matrix: Water

Date Received: 01/07/20 09:45

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1
DONA	ND		1.8		ng/L		01/15/20 05:09	01/16/20 07:38	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*107*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C4 PFHpA	*103*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C4 PFOA	*100*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C5 PFNA	*93*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C2 PFDA	*96*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C2 PFUnA	*98*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C2 PFDoA	*98*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C2 PFTeDA	*85*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C3 PFBS	*113*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
18O2 PFHxS	*112*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C4 PFOS	*110*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
d3-NMeFOSAA	*116*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
d5-NEtFOSAA	*126*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*
13C3 HFPO-DA	*100*		*25 - 150*	*01/15/20 05:09*	*01/16/20 07:38*	*1*

Client Sample ID: MW-6

Lab Sample ID: 320-57452-7

Date Collected: 01/03/20 13:54

Matrix: Water

Date Received: 01/07/20 09:45

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	**63**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluoroheptanoic acid (PFHpA)	**34**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorooctanoic acid (PFOA)	**36**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorononanoic acid (PFNA)	**5.5**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorodecanoic acid (PFDA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluoroundecanoic acid (PFUnA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorododecanoic acid (PFDoA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorotridecanoic acid (PFTriA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorobutanesulfonic acid (PFBS)	**14**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorohexanesulfonic acid (PFHxS)	**98**		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
Perfluorooctanesulfonic acid (PFOS)	**490**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		19		ng/L		01/15/20 05:09	01/16/20 07:46	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		19		ng/L		01/15/20 05:09	01/16/20 07:46	1
ADONA	ND		2.0		ng/L		01/15/20 05:09	01/16/20 07:46	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
HFPO-DA (GenX)	ND		3.8		ng/L		01/15/20 05:09	01/16/20 07:46	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1
DONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:46	1

DRAFT

Client Sample ID: MW-6

Date Collected: 01/03/20 13:54
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-7
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
13C2 PFHxA	69		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C4 PFHpA	67		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C4 PFOA	64		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C5 PFNA	63		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C2 PFDA	53		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C2 PFUnA	54		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C2 PFDoA	49		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C2 PFTeDA	35		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C3 PFBS	72		25 - 150	01/15/20 05:09	01/16/20 07:46	1
18O2 PFHxS	71		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C4 PFOS	66		25 - 150	01/15/20 05:09	01/16/20 07:46	1
d3-NMeFOSAA	67		25 - 150	01/15/20 05:09	01/16/20 07:46	1
d5-NEtFOSAA	67		25 - 150	01/15/20 05:09	01/16/20 07:46	1
13C3 HFPO-DA	49		25 - 150	01/15/20 05:09	01/16/20 07:46	1

Client Sample ID: MW-6-1

Date Collected: 01/03/20 13:57
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-8
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	**73**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluoroheptanoic acid (PFHpA)	**41**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorooctanoic acid (PFOA)	**40**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorononanoic acid (PFNA)	**6.5**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorodecanoic acid (PFDA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluoroundecanoic acid (PFUnA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorododecanoic acid (PFDoA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorotridecanoic acid (PFTriA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorotetradecanoic acid (PFTeA)	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorobutanesulfonic acid (PFBS)	**16**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorohexanesulfonic acid (PFHxS)	**110**		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
Perfluorooctanesulfonic acid (PFOS)	**540**	**E**	1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		19		ng/L		01/15/20 05:09	01/16/20 07:54	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		19		ng/L		01/15/20 05:09	01/16/20 07:54	1
ADONA	ND		2.0		ng/L		01/15/20 05:09	01/16/20 07:54	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
HFPO-DA (GenX)	ND		3.8		ng/L		01/15/20 05:09	01/16/20 07:54	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1
DONA	ND		1.9		ng/L		01/15/20 05:09	01/16/20 07:54	1

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
13C2 PFHxA	69		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C4 PFHpA	66		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C4 PFOA	66		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C5 PFNA	60		25 - 150	01/15/20 05:09	01/16/20 07:54	1

DRAFT

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Client Sample ID: MW-6-1
Date Collected: 01/03/20 13:57
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-8
Matrix: Water

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
13C2 PFDA	57		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C2 PFUnA	52		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C2 PFDoA	51		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C2 PFTeDA	36		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C3 PFBS	73		25 - 150	01/15/20 05:09	01/16/20 07:54	1
18O2 PFHxS	75		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C4 PFOS	68		25 - 150	01/15/20 05:09	01/16/20 07:54	1
d3-NMeFOSAA	69		25 - 150	01/15/20 05:09	01/16/20 07:54	1
d5-NEtFOSAA	68		25 - 150	01/15/20 05:09	01/16/20 07:54	1
13C3 HFPO-DA	56		25 - 150	01/15/20 05:09	01/16/20 07:54	1

DRAFT

Isotope Dilution Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Method: 537 (modified) - Fluorinated Alkyl Substances

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	PFHxA (25-150)	PFHpA (25-150)	PFOA (25-150)	PFNA (25-150)	PFDA (25-150)	PFUnA (25-150)	PFDoA (25-150)	PFTDA (25-150)
320-57452-1	MW-3	68	68	67	66	57	59	52	24 *
320-57452-2	MW-4	65	64	67	81	82	80	80	54
320-57452-3	MW-5	63	59	60	60	53	43	45	33
320-57452-4	MW-1	74	73	74	69	60	64	61	44
320-57452-5	IR-1	105	103	96	95	95	87	93	71
320-57452-6	ER-1	107	103	100	93	96	98	98	85
320-57452-7	MW-6	69	67	64	63	53	54	49	35
320-57452-8	MW-6-1	69	66	66	60	57	52	51	36
LCS 320-350842/2-A	Lab Control Sample	108	106	98	95	95	88	97	93
LCSD 320-350842/3-A	Lab Control Sample Dup	114	107	103	106	79	94	100	78
MB 320-350842/1-A	Method Blank	107	104	98	89	90	90	86	79

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	3C3-PFBS (25-150)	PFHxS (25-150)	PFOS (25-150)	-NMeFOS (25-150)	-NEtFOS (25-150)	HFPODA (25-150)		
320-57452-1	MW-3	80	76	73	67	70	38		
320-57452-2	MW-4	87	78	99	95	97	51		
320-57452-3	MW-5	67	65	62	61	64	56		
320-57452-4	MW-1	82	78	76	74	77	57		
320-57452-5	IR-1	105	108	105	110	112	73		
320-57452-6	ER-1	113	112	110	116	126	100		
320-57452-7	MW-6	72	71	66	67	67	49		
320-57452-8	MW-6-1	73	75	68	69	68	56		
LCS 320-350842/2-A	Lab Control Sample	110	109	105	115	114	70		
LCSD 320-350842/3-A	Lab Control Sample Dup	116	114	111	115	116	84		
MB 320-350842/1-A	Method Blank	108	111	107	110	114	88		

Surrogate Legend

PFHxA = 13C2 PFHxA
PFHpA = 13C4 PFHpA
PFOA = 13C4 PFOA
PFNA = 13C5 PFNA
PFDA = 13C2 PFDA
PFUnA = 13C2 PFUnA
PFDoA = 13C2 PFDoA
PFTDA = 13C2 PFTeDA
13C3-PFBS = 13C3 PFBS
PFHxS = 18O2 PFHxS
PFOS = 13C4 PFOS
d3-NMeFOSAA = d3-NMeFOSAA
d5-NEtFOSAA = d5-NEtFOSAA
HFPODA = 13C3 HFPO-DA

DRAFT

Method: 537 (modified) - Fluorinated Alkyl Substances

Lab Sample ID: MB 320-350842/1-A
Matrix: Water
Analysis Batch: 351059

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 350842

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorohexanoic acid (PFHxA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluoroheptanoic acid (PFHpA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorooctanoic acid (PFOA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorononanoic acid (PFNA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorodecanoic acid (PFDA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluoroundecanoic acid (PFUnA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorododecanoic acid (PFDoA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorotridecanoic acid (PFTriA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorotetradecanoic acid (PFTeA)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorobutanesulfonic acid (PFBS)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorohexanesulfonic acid (PFHxS)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
Perfluorooctanesulfonic acid (PFOS)	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
N-methylperfluorooctanesulfonamidoacetic acid	ND		20		ng/L		01/15/20 05:09	01/16/20 04:25	1
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND		20		ng/L		01/15/20 05:09	01/16/20 04:25	1
ADONA	ND		2.1		ng/L		01/15/20 05:09	01/16/20 04:25	1
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
HFPO-DA (GenX)	ND		4.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1
DONA	ND		2.0		ng/L		01/15/20 05:09	01/16/20 04:25	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C2 PFHxA	*107*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C4 PFHpA	*104*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C4 PFOA	*98*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C5 PFNA	*89*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C2 PFDA	*90*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C2 PFUnA	*90*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C2 PFDoA	*86*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C2 PFTeDA	*79*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C3 PFBS	*108*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
18O2 PFHxS	*111*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C4 PFOS	*107*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
d3-NMeFOSAA	*110*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
d5-NEtFOSAA	*114*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*
13C3 HFPO-DA	*88*		*25 - 150*	*01/15/20 05:09*	*01/16/20 04:25*	*1*

Lab Sample ID: LCS 320-350842/2-A
Matrix: Water
Analysis Batch: 351059

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 350842

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Perfluorohexanoic acid (PFHxA)	40.0	38.1		ng/L		95	73 - 133
Perfluoroheptanoic acid (PFHpA)	40.0	40.1		ng/L		100	72 - 132
Perfluorooctanoic acid (PFOA)	40.0	38.7		ng/L		97	70 - 130
Perfluorononanoic acid (PFNA)	40.0	41.4		ng/L		103	75 - 135
Perfluorodecanoic acid (PFDA)	40.0	38.8		ng/L		97	76 - 136

DRAFT

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCS 320-350842/2-A
Matrix: Water
Analysis Batch: 351059

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 350842

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Perfluoroundecanoic acid (PFUnA)	40.0	37.1		ng/L		93	68 - 128
Perfluorododecanoic acid (PFDoA)	40.0	36.2		ng/L		91	71 - 131
Perfluorotridecanoic acid (PFTriA)	40.0	39.2		ng/L		98	71 - 131
Perfluorotetradecanoic acid (PFTeA)	40.0	37.0		ng/L		92	70 - 130
Perfluorobutanesulfonic acid (PFBS)	35.4	36.0		ng/L		102	67 - 127
Perfluorohexanesulfonic acid (PFHxS)	36.4	35.1		ng/L		96	59 - 119
Perfluorooctanesulfonic acid (PFOS)	37.1	38.6		ng/L		104	70 - 130
N-methylperfluorooctanesulfonamidoacetic acid	40.0	35.9		ng/L		90	76 - 136
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	40.0	35.1		ng/L		88	76 - 136
ADONA	39.5	44.6		ng/L		113	79 - 139
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	37.3	39.6		ng/L		106	75 - 135
HFPO-DA (GenX)	40.0	54.0		ng/L		135	51 - 173
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	37.7	36.8		ng/L		98	54 - 114
DONA	37.7	42.5		ng/L		113	79 - 139

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
13C2 PFHxA	*108*		*25 - 150*
13C4 PFHpA	*106*		*25 - 150*
13C4 PFOA	*98*		*25 - 150*
13C5 PFNA	*95*		*25 - 150*
13C2 PFDA	*95*		*25 - 150*
13C2 PFUnA	*88*		*25 - 150*
13C2 PFDoA	*97*		*25 - 150*
13C2 PFTeDA	*93*		*25 - 150*
13C3 PFBS	*110*		*25 - 150*
18O2 PFHxS	*109*		*25 - 150*
13C4 PFOS	*105*		*25 - 150*
d3-NMeFOSAA	*115*		*25 - 150*
d5-NEtFOSAA	*114*		*25 - 150*
13C3 HFPO-DA	*70*		*25 - 150*

Lab Sample ID: LCSD 320-350842/3-A
Matrix: Water
Analysis Batch: 351059

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 350842

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Perfluorohexanoic acid (PFHxA)	40.0	39.5		ng/L		99	73 - 133	3	30
Perfluoroheptanoic acid (PFHpA)	40.0	40.6		ng/L		102	72 - 132	1	30
Perfluorooctanoic acid (PFOA)	40.0	36.8		ng/L		92	70 - 130	5	30
Perfluorononanoic acid (PFNA)	40.0	40.5		ng/L		101	75 - 135	2	30
Perfluorodecanoic acid (PFDA)	40.0	47.7		ng/L		119	76 - 136	21	30

DRAFT

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCSD 320-350842/3-A
Matrix: Water
Analysis Batch: 351059

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 350842

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Perfluoroundecanoic acid (PFUnA)			40.0	37.1		ng/L		93	68 - 128	0	30
Perfluorododecanoic acid (PFDoA)			40.0	33.6		ng/L		84	71 - 131	8	30
Perfluorotridecanoic acid (PFTriA)			40.0	37.6		ng/L		94	71 - 131	4	30
Perfluorotetradecanoic acid (PFTeA)			40.0	47.2		ng/L		118	70 - 130	24	30
Perfluorobutanesulfonic acid (PFBS)			35.4	36.5		ng/L		103	67 - 127	1	30
Perfluorohexanesulfonic acid (PFHxS)			36.4	34.7		ng/L		95	59 - 119	1	30
Perfluorooctanesulfonic acid (PFOS)			37.1	38.4		ng/L		104	70 - 130	1	30
N-methylperfluorooctanesulfonamidoacetic acid			40.0	39.2		ng/L		98	76 - 136	9	30
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)			40.0	37.2		ng/L		93	76 - 136	6	30
ADONA			39.5	43.0		ng/L		109	79 - 139	4	30
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid			37.3	39.2		ng/L		105	75 - 135	1	30
HFPO-DA (GenX)			40.0	47.5		ng/L		119	51 - 173	13	30
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid			37.7	36.8		ng/L		98	54 - 114	0	30
DONA			37.7	41.0		ng/L		109	79 - 139	4	30

Isotope Dilution	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
13C2 PFHxA	*114*		*25 - 150*
13C4 PFHpA	*107*		*25 - 150*
13C4 PFOA	*103*		*25 - 150*
13C5 PFNA	*106*		*25 - 150*
13C2 PFDA	*79*		*25 - 150*
13C2 PFUnA	*94*		*25 - 150*
13C2 PFDoA	*100*		*25 - 150*
13C2 PFTeDA	*78*		*25 - 150*
13C3 PFBS	*116*		*25 - 150*
18O2 PFHxS	*114*		*25 - 150*
13C4 PFOS	*111*		*25 - 150*
d3-NMeFOSAA	*115*		*25 - 150*
d5-NEtFOSAA	*116*		*25 - 150*
13C3 HFPO-DA	*84*		*25 - 150*

DRAFT

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

LCMS

Prep Batch: 350842

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-57452-1	MW-3	Total/NA	Water	3535	
320-57452-2	MW-4	Total/NA	Water	3535	
320-57452-3	MW-5	Total/NA	Water	3535	
320-57452-4	MW-1	Total/NA	Water	3535	
320-57452-5	IR-1	Total/NA	Water	3535	
320-57452-6	ER-1	Total/NA	Water	3535	
320-57452-7	MW-6	Total/NA	Water	3535	
320-57452-8	MW-6-1	Total/NA	Water	3535	
MB 320-350842/1-A	Method Blank	Total/NA	Water	3535	
LCS 320-350842/2-A	Lab Control Sample	Total/NA	Water	3535	
LCSD 320-350842/3-A	Lab Control Sample Dup	Total/NA	Water	3535	

Analysis Batch: 351059

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
320-57452-1	MW-3	Total/NA	Water	537 (modified)	350842
320-57452-2	MW-4	Total/NA	Water	537 (modified)	350842
320-57452-3	MW-5	Total/NA	Water	537 (modified)	350842
320-57452-4	MW-1	Total/NA	Water	537 (modified)	350842
320-57452-5	IR-1	Total/NA	Water	537 (modified)	350842
320-57452-6	ER-1	Total/NA	Water	537 (modified)	350842
320-57452-7	MW-6	Total/NA	Water	537 (modified)	350842
320-57452-8	MW-6-1	Total/NA	Water	537 (modified)	350842
MB 320-350842/1-A	Method Blank	Total/NA	Water	537 (modified)	350842
LCS 320-350842/2-A	Lab Control Sample	Total/NA	Water	537 (modified)	350842
LCSD 320-350842/3-A	Lab Control Sample Dup	Total/NA	Water	537 (modified)	350842

DRAFT

Lab Chronicle

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Client Sample ID: MW-3
Date Collected: 01/03/20 10:48
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-1
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			259.4 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 06:42	RS1	TAL SAC

Client Sample ID: MW-4
Date Collected: 01/03/20 11:20
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-2
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			259.6 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 06:50	RS1	TAL SAC

Client Sample ID: MW-5
Date Collected: 01/03/20 11:58
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-3
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			275.5 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 06:58	RS1	TAL SAC

Client Sample ID: MW-1
Date Collected: 01/03/20 12:37
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-4
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			279.4 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 07:06	RS1	TAL SAC

Client Sample ID: IR-1
Date Collected: 01/03/20 13:30
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-5
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			274.8 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 07:14	RS1	TAL SAC

Client Sample ID: ER-1
Date Collected: 01/03/20 13:35
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-6
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			280.4 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 07:38	RS1	TAL SAC

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Lab Chronicle

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Client Sample ID: MW-6

Date Collected: 01/03/20 13:54
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-7

Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			264.3 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 07:46	RS1	TAL SAC

Client Sample ID: MW-6-1

Date Collected: 01/03/20 13:57
Date Received: 01/07/20 09:45

Lab Sample ID: 320-57452-8

Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dil Factor	Initial Amount	Final Amount	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3535			261.9 mL	10 mL	350842	01/15/20 05:09	AF	TAL SAC
Total/NA	Analysis	537 (modified)		1			351059	01/16/20 07:54	RS1	TAL SAC

Laboratory References:

TAL SAC = Eurofins TestAmerica, Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

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Accreditation/Certification Summary

Client: Cox Environmental Services
Job ID: 320-57452-1
Project/Site: PFAS Compounds

Laboratory: Eurofins TestAmerica, Sacramento

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State	17-020	01-20-21
ANAB	Dept. of Defense ELAP	L2468	01-20-21
ANAB	Dept. of Energy	L2468.01	01-20-21
ANAB	ISO/IEC 17025	L2468	01-20-21
Arizona	State	AZ0708	08-11-20
Arkansas DEQ	State	19-042-0	06-17-20
California	State	2897	01-31-20 *
Colorado	State	CA0004	08-31-20
Connecticut	State	PH-0691	06-30-21
Florida	NELAP	E87570	06-30-20
Georgia	State	4040	01-29-20 *
Hawaii	State	<cert No.>	01-29-20 *
Illinois	NELAP	200060	03-17-20
Kansas	NELAP	E-10375	10-31-20 *
Louisiana	NELAP	01944	06-30-20
Maine	State	2018009	04-14-20
Michigan	State	9947	01-29-20 *
Michigan	State Program	9947	01-31-20
Nevada	State	CA000442020-1	07-31-20
New Hampshire	NELAP	2997	04-18-20
New Jersey	NELAP	CA005	06-30-20
New York	NELAP	11666	04-01-20
Oregon	NELAP	4040	01-29-20 *
Pennsylvania	NELAP	68-01272	03-31-20
Texas	NELAP	T104704399-19-13	05-31-20
US Fish & Wildlife	US Federal Programs	58448	07-31-20
USDA	US Federal Programs	P330-18-00239	07-31-21
Utah	NELAP	CA000442019-01	02-29-20
Vermont	State	VT-4040	04-16-20
Virginia	NELAP	460278	03-14-20
Washington	State	C581	05-05-20
West Virginia (DW)	State	9930C	12-31-19 *
West Virginia (DW)	State	9930C	12-31-20
Wyoming	State Program	8TMS-L	01-28-19 *

Laboratory: Eurofins TestAmerica, Seattle

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State	17-024	01-19-22
ANAB	Dept. of Defense ELAP	L2236	01-19-22
ANAB	ISO/IEC 17025	L2236	01-19-22
California	State	2901	11-05-20
Montana (UST)	State	NA	04-13-21
Oregon	NELAP	WA100007	11-06-20
US Fish & Wildlife	US Federal Programs	058448	07-31-20
USDA	US Federal Programs	P330-17-00039	02-10-20
Washington	State	C553	02-17-20

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

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Method Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Method	Method Description	Protocol	Laboratory
537 (modified)	Fluorinated Alkyl Substances	EPA	TAL SAC
3535	Solid-Phase Extraction (SPE)	SW846	TAL SAC

Protocol References:

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

Laboratory References:

TAL SAC = Eurofins TestAmerica, Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

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Sample Summary

Client: Cox Environmental Services
Project/Site: PFAS Compounds

Job ID: 320-57452-1

Lab Sample ID	Client Sample ID	Matrix	Collected	Received	Asset ID
320-57452-1	MW-3	Water	01/03/20 10:48	01/07/20 09:45	
320-57452-2	MW-4	Water	01/03/20 11:20	01/07/20 09:45	
320-57452-3	MW-5	Water	01/03/20 11:58	01/07/20 09:45	
320-57452-4	MW-1	Water	01/03/20 12:37	01/07/20 09:45	
320-57452-5	IR-1	Water	01/03/20 13:30	01/07/20 09:45	
320-57452-6	ER-1	Water	01/03/20 13:35	01/07/20 09:45	
320-57452-7	MW-6	Water	01/03/20 13:54	01/07/20 09:45	
320-57452-8	MW-6-1	Water	01/03/20 13:57	01/07/20 09:45	

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TestAmerica Anchorage
2000 W. International Airport Road
Suite A10
Anchorage, AK 99502
Phone: 907.563.9200 Fax: 907.563.9210

Chain of Custody Record 249144

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Laboratories, Inc.
TAL-8210 (0713)

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

Client Contact	Project Manager: J. COX	Site Contact: J. COX	Date: 1.3.20	COC No: 1
Company Name: CH2M HILL	Tel/Fax: 907.723.9946	Lab Contact: S. COX	Carrier: AK AIR	1 of 1 COCs
Address: 2525 C ST, SUITE 300	Analysis Turnaround Time			Sampler: J. COX
City/State/Zip: ANCHORAGE, AK 99503	☒ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
Phone: 907.564.4447	TAT if different from Below			Walk-in Client:
Fax:	☒ 2 weeks			Lab Sampling:
Project Name: JIA PFAS	☐ 1 week			Job / SDG No.:
Site:	☐ 2 days			
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)	PFAS 537	Sample Specific Notes:
MW-3	1.3.20	10:48	G	W	2			X	
MW-4		11:20						X	
MW-5		11:58						X	
MW-1		12:37						X	
12-1		13:30						X	
EB-1		13:35						X	
MW-6		13:54						X	
MW-6-1		13:57						X	

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320-57452 Chain of Custody

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.: 890494 890493	Cooler Temp. (°C): Obs'd: 5.2 Corr'd: 5.2	Therm ID No.: IR/Stick

Relinquished by:	Company: CH2M	Date/Time: 1/3/20 16:58	Received by: Juan Guzm	Company: ETA-SAC	Date/Time: 1/7/19/1138
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Login Sample Receipt Checklist

Client: Cox Environmental Services

Job Number: 320-57452-1

Login Number: 57452
List Number: 1
Creator: Kintaudi, Pauline W

List Source: Eurofins TestAmerica, Sacramento

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	890494/890493
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

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ATTACHMENT C

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by JMC date 11/13/19 client CB&J

description JIA PFAS WELL INSTALLATION

11/13/19 JIA PFAS WELL INSTALLATION RAIN 45° LIGHT WINDS

0930 ARRIVE ON-SITE MEET W/ AARON DEAN

LOCATE POTABLE WATER SOURCE

LOCATE MW-3

1030 BEGIN SETUP ; DECON EQUIPMENT

11 10 BEGIN SETUP ON MW-2

JIA-MW-2

JIA-EB-1 11:15 RINSE OF SPLIT SPOON, TOOLING

PFAS 537

JIA-MW-2 H_2O @ 4.2'

PFAS 537 12:16 SAMPLE DEPTH 4'

% MOISTURE

WELL SET @ 13:30 WELL TO BE SET @ 3'

DECON / MOVE TO MW-3

15:40

BEGIN DRILLING MW-3

JIA-MW-3 16:14 SAMPLE DEPTH 4.5'

PFAS 537 H_2O @ 4.5'

% MOISTURE WELL TO BE SET @ 3.5'

WELL SET @ 17:35

DECON / DEMOBILIZATION

LEFT SITE @ 18:40

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by date client

descriptic

11/14/19 JIA PFAS

RAIN 40's WINDY

ON-SITE 09:00

BEGIN SITE SET-UP @ MW-4

DECON EQUIPMENT

BEGIN DRILLING @ 10:40 JIA-IR-1 11:10

H_2O RINSE OF SAMPLE SPOON

PFAS 537

JIA-MW-4

PFAS 537

% MOISTURE 12:10

WATER @ 6.5'

WELL SET @ 9.5'

BEGIN DECON @ 1:45, MOVE TO MW-5

NOTIFIED MIKE GREEN, NEED TO CONTACT ATC

NOTAM REQUIRED to DRILL @ MW-5 WITHIN RSA.

MOVED EQUIPMENT TO MW-6

LEFT SITE @ 15:35

11/15/19

ON-SITE @ 09:00

SETUP / DECON EQUIPMENT

09:45 BEGIN DRILLING @ MW-6

JIA-MW-6

JIA-MW-6-1 (DUP)

PFAS 537

% moist

H_2O @ 8.5' WELL SET 7-12'

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by date client

description

DECON/MOVE TO MW-1
LEFT SITE @ 14:00

11/16/19 JIA PFAS
ON-SITE 08:30
BEGIN SITE SET-UP & DECON
BEGIN DRILLING @ MW-1 09:30

JIA-EB-2 10:00
RINSE SPLIT SPOON/ROD

JIA-MW-1 13:52
PFAS 53.7
% MOIST

H_2O @ 13'
WELL SET 16'-11'
14:50 BEGIN DECON/DEMOB
15:20 LEFT SITE

11/18/19
08:30 ON-SITE
MEET SCOTT RINKENBERGER
ESCORT (FRANK) NOTAM ACTIVE
08:45 MOVE TO DRILL LOCATION
09:00 BEGIN DECON/SETUP @ MW-5
09:15 BEGIN DRILLING
H_2O @ 6.5' WELL SET 4-9'

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by _____ date _____ client _____

description _____

11:00 DECON & DEMOB

11:55 OFF-SITE

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SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-1	JIA PFAS	

Boring Depth (ft) x Diameter (in):	Drilling Contractor Name:	Drilling Method:
16' x 8"	DENALI	AUGER - SPLIT

Date Start Time:	Date End Time:	Logged By: M.
11.14.19 09:30	14:50	J. COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2	 - BENTONITE TO SURFACE					
2/4						
4/6					Silt 6-7' lt. brown p. graded sand 7'-8'	
6/8	10' - SAND				lt. brown poorly graded sand	
8/10	11'				lt. brown poorly graded sand	
10/12	13'				lt. brown poorly graded sand	
14/16	16'	JIA-MW-1 PFAS 537 % moist 13		13:52	thin layer silt @14 lt. brown p. graded sand	

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SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-2	JIA PFAS	

Boring Depth (ft) x Diameter (in):	Drilling Contractor Name:	Drilling Method:
8.5' x 6"	DENALI	AUGER/SPLIT

Date Start Time:	Date End Time:	Logged By: M.
11:10 11.13.19	13:30	J. COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2	 BENT-SURF 2' SAND					
2/4	3' 4'					
4/6		JIA-MW-2 5314 / 8% MOIST 12:16 4'			GRAY POORLY GRADED SAND	
4/8	8'				GRAY POORLY GRADED SAND GRAY SILT 7'	

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SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-3	JIA PFAS	

Boring Depth (ft) x Diameter (in):	Drilling Contractor Name:	Drilling Method:
8.5' x 6"	DENALI	AUGER/SPLIT

Date Start Time:	Date End Time:	Logged By: M.
15:40 11/13/19 ~~17:35~~	17:35	J.COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2						
2/4						
4/4		JIA-MW-3 55% moist 4.5'		16:14	GRAY POORLY GRADED SAND	
4/8.5					GRAY POORLY GRADED SAND GRAY SILT 6'	

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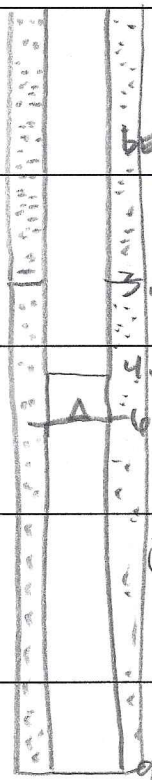
SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-4	JIA PFAS	

Boring Depth (ft) x Diameter (in):	Drilling Contractor Name:	Drilling Method:
8' x 6"	DENALI	AUGER / SPLIT SPOON

Date Start Time:	Date End Time:	Logged By: M.
11/14/19 10:40	13:45	J. COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2	 bentonite to surface					
2/4	3.5' SAND				lt. br C. P. GRADED SAND	
4/4	4.5' TO SCREEN 6.5' H_2O				DK. GRAY SILT	
6/8		JIA - MW-4 537 PFAS 12:10 % moist 4.5'			lt. gray poorly graded SAND COARSE	
8/10	9.5					

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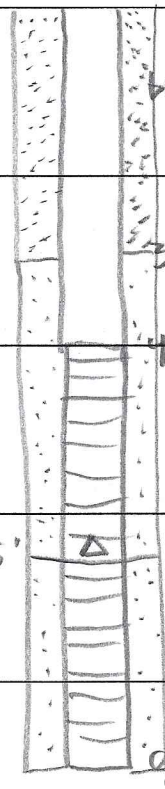
SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-5	JIA PFAS	
Boring Depth (ft) x Diameter (in):	**Drilling Contractor Name:**	**Drilling Method:**
9' x 6"	DENALI	AUGER-SPOON
Date Start Time:	**Date End Time:**	**Logged By: M.**
11.18.19 09:15	11.18.19 10:54	J. COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2						
2/4						
4/6					lt. brown poorly graded coarse sand	
4/8	JIA-MW-5 PFAS 537 % moist	13:52 6.5'			lt. brown poorly graded coarse sand	
8/10						

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SOIL BORING LOG & MONITORING WELL CONSTRUCTION DIAGRAM

Boring/Well #:	Site Name:	Site Address:
MW-6	JIA PFAS	

Boring Depth (ft) x Diameter (in):	Drilling Contractor Name:	Drilling Method:
13.5 x 6"	DENALI	AUGER - SPOON

Date Start Time:	Date End Time:	Logged By: M.
11.15.19 09:45	11.15.19 13:10	J. COX

Depth (ft)	Well Construction Details	Sample ID	Analytical Methods	PID Readings	Soil Classification & Observations	Soil Graphic
0/2						
2/4						
4/6					silt gray	
4/8					silt gray	
8/10		8.5' JIA-MW-6 12:17 JIA-MW-6-1 12:19 PFAS 537 (DUPLICATE) % moist			silt gray 8.5 - C. SAND	
10/12					lt. gray COARSE WELL GRADED SAND	
12/16						

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WELL DEVELOPMENT PURGE AND SAMPLING FIELD SHEET

cox environmental
712 W 12th Street Juneau, AK 99801
907-586-4447

Project:	Juneau International Airport PFAS	Well ID	MW-1
		Date:	11/3/20
		Start Time:	12:15
Sampler	J. COX	End Time:	12:36
Sample ID	MW-1 PFAS 537	Time:	12:37
Sample ID		Time:	

Weather Conditions:

Depth to Top of Product (FTOC):			Depth to Water (FTOC):	12.6
Depth to Oil/Water Interface (FTOC):			Total Depth (FTOC):	13.7
Casing Diameter	2 in.	4 in.	Water Column (Ft):	1.1
G/Ft of Casing	0.16	0.65	Casing Volume (Gallons)	0.17

Method of Purge & Sampling: Peristaltic Pump

Start Time:	12:15
End Time:	12:36
Vol. Purged (gals):	0.5

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WELL DEVELOPMENT PURGE AND SAMPLING FIELD SHEET

cox environmental
712 W 12th Street Juneau, AK 99801
907-586-4447

				Well ID	MW-3
Project:	Juneau International Airport PFAS			Date:	1/3/19
				Start Time:	10:20
Sampler	J. COX			End Time:	10:48
Sample ID	MW-3 PFAS 537	Time:	10:48		
Sample ID		Time:			

Weather Conditions:

Depth to Top of Product (FTOC):			Depth to Water (FTOC):	7.9
Depth to Oil/Water Interface (FTOC):			Total Depth (FTOC):	10.9
Casing Diameter	2 in.	4 in.	Water Column (Ft):	3.0
G/Ft of Casing	0.16	0.65	Casing Volume (Gallons)	0.48

Method of Purge & Sampling: Peristaltic Pump

Start Time:	10:20
End Time:	10:48
Vol. Purged (gals):	1.5

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WELL DEVELOPMENT PURGE AND SAMPLING FIELD SHEET

cox environmental
712 W 12th Street Juneau, AK 99801
907-586-4447

Project:	Juneau International Airport PFAS	Well ID	MW-4
		Date:	1.3.19
		Start Time:	11:02
Sampler	J.COX	End Time:	11:20
Sample ID	MW-4 537 PFAS	Time:	11:20
Sample ID		Time:	

Weather Conditions:

Depth to Top of Product (FTOC):			Depth to Water (FTOC):	8.9
Depth to Oil/Water Interface (FTOC):			Total Depth (FTOC):	12.7
Casing Diameter	2 in.	4 in.	Water Column (Ft):	3.8
G/Ft of Casing	0.16	0.65	Casing Volume (Gallons)	0.6

Method of Purge & Sampling: Peristaltic Pump

Start Time:	11:02
End Time:	11:20
Vol. Purged (gals):	2

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WELL DEVELOPMENT PURGE AND SAMPLING FIELD SHEET

cox environmental
712 W 12th Street Juneau, AK 99801
907-586-4447

				Well ID	MW-5
Project:	Juneau International Airport PFAS			Date:	1.3.20
				Start Time:	11:46
Sampler	J. COX			End Time:	11:58
Sample ID	MW-5	Time:	11:58		
Sample ID		Time:			

Weather Conditions:

Depth to Top of Product (FTOC):			Depth to Water (FTOC):	9.6
Depth to Oil/Water Interface (FTOC):			Total Depth (FTOC):	12.1
Casing Diameter	2 in.	4 in.	Water Column (Ft):	2.5
G/Ft of Casing	0.16	0.65	Casing Volume (Gallons)	0.4

Method of Purge & Sampling: Peristaltic Pump

Start Time:	11:40
End Time:	11:58
Vol. Purged (gals):	1.2

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WELL DEVELOPMENT PURGE AND SAMPLING FIELD SHEET

cox environmental
712 W 12th Street Juneau, AK 99801
907-586-4447

Project:	Juneau International Airport PFAS	Well ID	MW-6
		Date:	1.3.20
		Start Time:	13:30
Sampler		End Time:	13:57
Sample ID	MW-6	Time:	13:54
Sample ID	MW-6-1	Time:	13:57

Weather Conditions:

Depth to Top of Product (FTOC):			Depth to Water (FTOC):	12.7
Depth to Oil/Water Interface (FTOC):			Total Depth (FTOC):	14.8
Casing Diameter	2 in.	4 in.	Water Column (Ft):	2.1
G/Ft of Casing	0.16	0.65	Casing Volume (Gallons)	0.3

Method of Purge & Sampling: Peristaltic Pump

Start Time:	13:30
End Time:	13:54
Vol. Purged (gals):	1.0

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ATTACHMENT D

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cox environmental

PFAS SAMPLING AND ANALYSIS PLAN (SAP)

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Juneau International Airport
1873 Shell Simmons Drive Juneau, Alaska

Prepared for:

City & Borough of Juneau

Submitted to:

Alaska Department of Environmental Conservation
Division of Spill Prevention and Response
Contaminated Sites Program
410 Willoughby Ave., Ste. 303
P.O. Box 111800
Juneau, AK 99811

August 2019

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ACRONYM LIST

ADEC	Alaska Department Of Environmental Conservation
AFFF	Aqueous Film-Forming Foams
ARFF	Aircraft Rescue and Fire-Fighting
ATSDR	Agency for Toxic Substances and Disease Registry
CBJ	City & Borough of Juneau
CES	Cox Environmental Services
CFR	Code of Federal Regulations
COPC	Constituents of Potential Concern
CSM	Conceptual Site Model
DO	Dissolved Oxygen
DOT	Department of Transportation
DPT	Direct Pushed Technology
ETFE	Ethylene Tetrafluoroethylene
FAA	Federal Aviation Administration
FEP	Fluorinated Ethylene Propylene
GAC	Granular Activated Carbon
HDPE	High-Density Polyethylene
IDW	Investigation-Derived Wastes
ITRC	Interstate Technology Regulatory Council
JIA	Juneau International Airport
LDPE	Low Density Polyethylene
LHA	Lifetime Health Advisory
MDL	Method Detection Limits
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSDS	Material Safety Data Sheets
ORP	Oxidation-Reduction Potential
PFAS	Per and Polyfluoroalkyl Substances
PFBS	Perfluorobutanesulfonic Acid
PFHpA	Perfluoroheptanoic Acid
PFHxS	Perfluorohexanesulfonic Acid
PFNA	Perfluorononanoic Acid
PFOA	Perfluoroctanoic Acid
PFOA	Perfluorooctanoic Acid
PFOS	Perfluorooctane Sulfonate
PFOS	Perfluorooctane Sulfonate
PPE	Personal Protective Equipment
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
PVDF	Polyvinylidene Fluoride
QA/QC	Quality Assurance Quality Control
RL	Reporting Limits
SAP	Sampling and Analysis Plan
USEPA	United States Environmental Protection Agency

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1 Introduction & Site Background

This Sampling and Analysis Plan (SAP) presents the objectives and strategies for soil and groundwater sampling activities at the Juneau International Airport (JIA). Cox Environmental Services (CES) has been contracted by the City & Borough of Juneau (CBJ) to provide environmental services in support of characterization of potential Per and Polyfluoroalkyl Substances (PFAS) contamination.

PFAS are a group of synthetic chemicals that have been in use since the 1940s. PFAS are found in a wide array of consumer and industrial products. PFAS manufacturing and processing facilities, facilities using PFAS in production of other products, airports, and military installations are some of the contributors of PFAS releases into the air, soil, and water. Due to their widespread use and persistence in the environment, most people in the United States have been exposed to PFAS. There is evidence that continued exposure above specific levels to certain PFAS may lead to adverse health effects (USEPA 2016a, 2016b, ATSDR 2018a).

Because PFAS are persistent in the environment and soluble in water, large plumes of groundwater contamination can form where these compounds have been released. When releases occur in areas served by private or public drinking water wells, the well water is susceptible to contamination. When PFAS contamination is found in the environment, the responsible party must evaluate the extent of the contamination in the soil and groundwater, determine whether and to what extent drinking water supplies are impacted, provide treatment or alternative water if action levels are exceeded, and begin cleanup with ADEC's oversight. The responsible party is typically the entity that caused the release or the landowner where the release occurred.

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In Alaska, spills or releases of PFAS into the environment are primarily associated with the use of aqueous film-forming foams (AFFF) during firefighting or fire training activities. PFAS compounds of concern where AFFF has been used include perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). Although these two compounds are the most studied, a growing body of research indicates additional PFAS compounds may have similar health or environmental effects and may be co-contaminants.

Title 14 Code of Federal Regulation (CFR) Part 139 requires airport operators to maintain their aircraft rescue and firefighting (ARFF) vehicles and their fire suppression operating systems. JIA is a Part 139 Airport. Such systems, including the foam proportioning system and discharge functions, must be able to operate properly in an emergency situation. To help ensure their operability, the Federal Aviation Administration (FAA) recommends vehicle system testing intervals occur within 6 months of the airport's

periodic airport certification safety inspection. Currently, all certificated Part 139 airports are required to use foams that meet military specifications (MIL-PRF-24385). The growing concern over the use and discharge of AFFF at airports has led to the inclusion of a mandate within the FAA Reauthorization Act of 2018 (enacted October 5, 2018), directing the FAA to stop requiring the use of fluorinated foam no later than three years from the date of enactment (October 4, 2021).

The airport currently uses CHEMGUARD C306-MS 3% AFFF, the formulation contains short-chain, C-6 fluorochemicals manufactured using a telomer-based process. The telomer process produces no PFOS, and these C-6 materials do not breakdown to yield PFOA. Historical releases of PFAS from AFFF during historical training activities are believed to have taken place in five locations at the airport, actual quantities of AFFF or dates of discharges at the site are not documented. The historical release locations are depicted on *Figure 5 Potential AFFF Discharge Areas.*

2 Site Description

The site is located at 1873 Shell Simmons Drive in Juneau, Alaska. The location of the site is depicted on *Figure 1. Site Location Map.* The features and details of the property are depicted on *Figure 2. 2013 Aerial Photograph* and *Figure 3. Site Plan.*

JIA is owned and operated by the CBJ. The Airport was originally developed by the United States government to support military Air Corps operations in Alaska. Prior to World War II, the area was served by a limited number of small aircraft, mostly float planes. The paved runway at the Airport was constructed in 1942. Following the war, Pan American Airlines and Pacific Northern Airlines established service to Juneau from Seattle and Anchorage. The original terminal was constructed in 1948. In 1953, the Airport was transferred from U.S. government ownership to the City of Juneau. The first of two major terminal expansions took place in 1957, and the second expansion, resulting in the Airport's present configuration, took place in 1984. The Airport has undergone other modifications as well. In 1961, the runway was extended to accommodate jet aircraft operations in Alaska. In 1989, a full-length parallel taxiway was constructed to connect both ends of the runway to the aircraft parking apron and passenger terminal area. Other facility improvements have taken place periodically, most recently for additional aircraft parking and hangar spaces.

2.1 Climate

Juneau is near the northern end of a temperate rain forest found on the North Pacific Coast from San Francisco to Anchorage. Juneau weather is characterized by a North Pacific maritime climate with

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frequent storms and abundant precipitation. The mean summer high temperature is 62° F, and the mean winter high temperature is 33° F. There are approximately 150 frost-free days annually.

2.2 Hydrology

The JIA is located on the Mendenhall River delta. Material excavated from the Float Plane Pond was used in the original airport construction (Adamus 1987) in the late 1930s and early 1940s, and a wide variety of other sources were used as fill to develop the large elevated surface that the JIA now occupies.

The JIA is bordered by the Mendenhall River on the west, the Mendenhall Wetlands Refuge to the south and east, and industrial and commercial land to the north. A dike in the western and southern portions of the airport property protects the Float Plane Pond and other assets from the Mendenhall River to the west and from the Gastineau Channel to the south and southeast. The Gastineau Channel inundates the refuge daily at high tide.

Duck Creek enters the airport property from the northwest, through a culvert under Berners Avenue. Duck Creek bends southwest through an undeveloped parcel of land in the northwest corner of the airport property. Duck Creek leaves the site via a culvert that passes under the dike to the west and discharges to the Mendenhall River approximately 1,500 feet later. The creek has been channelized in several reaches through infrastructure development activities. The floodplain is constricted in many locations.

Jordan Creek enters the airport property from the north approximately 1,400 feet east of Duck Creek. Jordan Creek crosses Yandukin Drive and meanders for approximately 1,300 feet before crossing underneath Crest Street through a culvert. Jordan Creek is channelized along portions of the reach below Yandukin Drive, and the floodplain is especially constricted below Crest Street. The channel bends sharply as it travels through the airport property. The creek passes through long culverts under the taxiway and then the runway prior to leaving the airport property and entering the refuge.

Vegetated ditches that drain most of the runways and taxiways discharge stormwater to Duck Creek and Jordan Creek. High tides create backwater conditions on Jordan Creek that can cause ponding in these ditches.

The Float Plane Pond is surrounded by the runway to the north, the Mendenhall River to the west, the dike to the south, and the mouth of Jordan Creek to the east. A dike separates the Float Plane Pond from tidal wetlands and the Gastineau Channel. The Float Plane Pond is approximately 5,300 feet long by 430 feet wide, with an average depth of four to five feet. A 30-foot deep pocket of water is located in the south end

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of the pond. Several sloughs and side channels extend from the main body of the pond into the wooded area to the south. The total surface area of the Float Plane Pond is approximately 80 acres, including sloughs and side channels. The water level of the pond is controlled by a tide gate at the west end of the pond. During high tide conditions, brackish water from the Mendenhall River enters the pond through this structure.

2.3 General Geology and Soils

The JIA is located on the northeast side of Gastineau Channel, within the Mendenhall River Basin, which extends from the Coast Mountains of southeast Alaska. Several studies have described the geology of the Mendenhall Valley (Alcorn and Hogan 1995, Barnwell and Boning 1968, Hicks and Shofnos 1965, Motyka 1988). The underlying bedrock is composed of tightly consolidated sedimentary (slate, greywacke, and sandstone), igneous (extruded volcanics), and metamorphic rocks (greenstone and schist) that are relatively impervious to moisture. The surficial geology in the area around the Gastineau Channel, the JIA, and Mendenhall Valley includes glaciomarine deposits of the Gastineau Channel Formation, overlain by glacial outwash deposits. The outwash deposits range in thickness from 10 feet to 100 feet. They are comprised of sand-size to cobble-size rocks that have been overlain in some small areas, mostly down the middle of the valley, by muskeg or plant debris in various stages of decay. Moraine deposits composed of loose till and unsorted gravelly sand are found in the upper valley. Farther down the valley, beach deposits and glaciomarine deposits from the Gastineau Channel Formation characterize most of the Gastineau Channel.

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The JIA property consists of the BeA, CoA, and LeA soil-mapping units, with BeA the predominant mapping unit on the JIA property (USDA 1974). BeA is excessively drained, very gravelly sand with 0% to 3% slopes. This soil is found on nearly level alluvial plains and terraces, along with spots of wet, sandy soils. This soil rarely floods. However, in a few low-lying areas near the coast and adjacent to streams, inundation may occur when tides or streams are exceptionally high. A small portion of the northwest JIA property consists of the CoA soil-mapping unit. This soil is a poorly drained silt loam found on low-lying, nearly level, alluvial plains. The angles of slopes on which this soil is found range from 0% to 3%. In most places, this soil is susceptible to occasional overflow from freshwater streams, and in a few places it may be inundated by exceptionally high tides. This soil unit may include spots of very poorly drained shallow peat soils. A small section on the northern edge of the JIA property consists of the LeA soil-mapping unit. This mapping unit includes areas of small streams. The soil is a very poorly drained silt loam found on slight depressions in broad stream valleys; the slope ranges from 0% to 3% and is almost always nearly level. This soil is susceptible to occasional flooding.

2.4 Groundwater

The Mendenhall Valley contains two aquifers (Barnwell and Boning 1968). The upper aquifer lies within the unconfined sediments of silt, sand, and gravel at a depth of 3 to 15 feet below the ground surface. The thickness of the upper aquifer ranges from 0 to 300 feet (Osgood 1990). Mendenhall Lake is a major source of recharge for the upper aquifer. The lower aquifer is separated from and confined by a layer of bedrock below the upper aquifer. The water table flows southwesterly through the valley towards the Mendenhall River.

The JIA is located on the Mendenhall River delta, and the runway and associated taxiways were built up by fills of sand and gravel, mostly from the excavation of the Float Plane Pond. The porous nature of the geology under the JIA, possibly including the old stream channels from Duck Creek and Jordan Creek, likely facilitates groundwater interaction with surface water.

3 Previous Investigations

No previous investigations related to PFAS have been conducted at the site.

4 Scope of Work

The general purpose of this SAP is to provide a detailed description of site characterization activities to be conducted at the site to delineate the extent of potential PFAS impacts to soil and groundwater from historical use of AFFF at the JIA.

The planned scope of work to be conducted under this SAP will generally consist of:

- Installation of 6 soil borings and 6 groundwater monitoring wells to evaluate potential impacts to soil and groundwater. The soil borings and monitoring wells will be drilled using Geoprobe direct-push drilling methods.
- Collection of soil and groundwater samples for laboratory analysis.
- Collection of groundwater samples from two of the existing 16 on-site groundwater wells. Monitoring well (MW-4) located at the airport Tank Farm and monitoring well (MW-10) located at the Alaska Airlines GSE/Air Cargo Facility.

The proposed soil borings, proposed groundwater monitoring well locations, and existing monitoring well locations are depicted on *Figure 4. Site Plan with Proposed Soil Borings and Groundwater Monitoring Well Locations.*

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5 Soil and Groundwater Constituents of Potential Concern

The following are the soil and groundwater constituents of potential concern (COPCs):

- PFAS
  - Perfluoroctanoic acid (PFOA) and Perfluorooctane sulfonate (PFOS)

6 Conceptual Site Model

In accordance with ADEC requirements, CES has completed a Conceptual Site Model (CSM) evaluation for the site. The CSM evaluation consisted of CES completing the ADEC Scoping form and ADEC Flow Chart Graphic, which are included in Attachment B. The CSM identifies the medium, transport mechanisms, exposure medium, exposure pathways/routes, current and future receptors, and whether each exposure pathway is complete currently, in the future, both currently and in the future, or insignificant. The CSM evaluation is based on the release of PFAS from required testing of AFFF equipment at the site and that no site assessment has occurred to date, so no analytical data are available.

6.1 Pathways of Exposure

An exposure pathway describes the course that a constituent takes from its environmental source to a receptor. Each exposure pathway includes the following elements: (1) a source or constituent release from a source (transport mechanism), (2) an exposure medium, (3) a point of potential contact for the receptor with the exposure medium, and (4) an exposure pathway/route at the contact point (e.g., ingestion, dermal contact or inhalation). An exposure pathway is considered complete when all of these elements are present.

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Once constituents are released into an environmental medium, they may migrate from one medium to another. Complete exposure pathways are those that involve receptor contact with an environmental medium that contains elevated levels of site-associated constituents.

6.1.1 Exposure Media and Routes of Exposure

Soil – Incidental Ingestion, Dermal Absorption, Inhalation:

For receptors with potential exposure to soil, incidental soil ingestion, dermal absorption of contaminants from soil, and inhalation of fugitive dust are the exposure pathways/routes evaluated in the CSM.

Groundwater –Ingestion, Dermal Absorption, Inhalation:

For receptors with potential exposure to groundwater, incidental ingestion of groundwater, dermal absorption of contaminants in groundwater, and inhalation of volatile constituents in tap water are the exposure pathways/routes evaluated in the CSM.

Air –Inhalation:

For receptors with potential exposure to air, inhalation of outdoor air, inhalation of indoor air, and inhalation of fugitive dust are the exposure pathways/routes evaluated in the CSM.

Surface Water –Ingestion, Dermal Absorption, Inhalation:

For receptors with potential exposure to surface water, incidental ingestion of surface water, dermal absorption of contaminants in surface water, and inhalation of volatile constituents in tap water are the exposure pathways/routes evaluated in the CSM.

Sediment –Direct Contact:

For receptors with potential exposure to sediment, direct contact with sediment is the exposure pathways/route evaluated in the CSM.

Biota –Ingestion:

For receptors with potential exposure to biota, ingestion of wild or farmed foods is the exposure pathways/route evaluated in the CSM.

6.1.2 Receptors

The potential current and future human receptors at a site must be characterized in order to evaluate potential exposure pathways. The CSM includes the following receptors:

- Residents (Adults or Children)
- Commercial or Industrial Workers
- Site Visitors, Trespassers, or Recreational Users
- Construction Workers
- Farmers or Subsistence Harvesters
- Subsistence Consumers
- Other

6.1.3 Potentially Complete Pathways of Exposure

The potentially complete exposure pathways for current and future receptors for the JIA are identified below. While the site is likely to remain a commercial property, potential future land use is assumed to also include the most conservative residential scenario.

Soil

Direct contact with soil is possible for commercial or industrial workers and construction workers involved in subsurface excavation activities or future residents in the most conservative residential scenario.

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Groundwater

Direct contact with groundwater is possible if commercial or industrial workers and construction workers involved in subsurface activities excavate down to the water table. Groundwater at the JIA and in the vicinity of the JIA is not currently used or likely to be used in the future as a source of drinking water. Drinking water is provided by the City & Borough of Juneau Municipal water supply. However, the pathway is considered complete for future residents in the most conservative residential scenario.

Outdoor Air

PFAS are not volatile constituents so this pathway is considered incomplete.

Indoor Air

PFAS are not volatile constituents so this pathway is considered incomplete.

Surface Water

On-site surface water drainages including Jordan Creek, Duck Creek, and the float pond discharge into the Mendenhall River, Gastineau Channel, and the Mendenhall Wetlands Reguge. Potential contamination in on-site soil and hydrologically connected groundwater may migrate to on-site and off-site surface water. Surface water at the site and in the vicinity of the site is not currently used or likely to be used in the future as a source of drinking water. However, the pathway is considered complete for future residents in the most conservative residential scenario.

Sediment

Direct contact with sediment is possible for commercial or industrial workers and construction workers involved in subsurface excavation activities or future residents in the most conservative residential scenario.

Biota

The adjacent Mendenhall Wetlands Refuge wetlands to the south and east of the JIA provide potential ecological habitat, so the ingestion of wild or farmed foods pathway is considered potentially complete. The terrestrial and aquatic ecological exposure pathways are also considered potentially complete.

7 Soil and Groundwater Analytical Methods

Soil and groundwater samples will be analyzed by our contract laboratory, Test America, Inc. Samples will be analyzed for:

- PFAS using Modified Method 537
    - Perfluorobutanesulfonic acid (PFBS)

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- Perfluorohexanesulfonic acid (PFHxS)
- Perfluoroheptanoic acid (PFHpA)
- Perfluoroctanoic acid (PFOA)
- Perfluorooctane sulfonate (PFOS)
- Perfluorononanoic acid (PFNA)

Analytical data validation will be performed by CES by filling out the ADEC Laboratory Data Review Checklist for each laboratory data deliverable.

8 Soil and Groundwater Sampling

Sampling conducted to determine PFAS concentrations in soil and groundwater is similar to that for other chemical compounds, but with several additional specific considerations and protocols. The soil and groundwater sampling activities will be performed in general accordance with following documents:

- ADEC's Field Sampling Guidance (ADEC August 2017)
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for PFAS, (Interstate Technology Regulatory Council's (ITRCs) March 2018)
- Bottle Selection and other Sampling Considerations When Sampling for PFAS (USDOD EDQW 2017b)
- Interim Guideline on the Assessment and Management of PFAS, Contaminated Sites Guidelines, (Government of Western Australia, Department of Environment Regulation 2016)
- Alpha Analytical EPA 537 (PFAS) Field Sampling Guidelines

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8.1 Soil Boring Installation and Soil Sampling

The soil borings with be advanced with a Geoprobe 6620DT and soil sampling will be performed using Direct Pushed Technology (DPT) and Geoprobe macrocore soil sampling tooling. One soil sample will be collected at each soil boring from the soil groundwater interface. CES personnel will use disposable polyethylene spoons to collect soil samples. Prior to handling any soil, CES personnel will don a new pair of disposable nitrile gloves which will be interchanged prior to collection of each soil sample. Soil samples to be analyzed for PFAS will be placed into laboratory certified unpreserved 4-oz HDPE jars. Soil samples for PFAS will be unpreserved. All sample jars will be labeled with the project name, sample identification number, date/time of sample collection, preservative, analysis requested, and sampler's initials. The samples will be kept in a sample cooler with double bagged ice (at 4°C ± 2°C) pending delivery to the contract laboratory. The soil borings will be installed and soil samples will be collected in accordance with CES Standard Operating Procedure SP-01 (SP-01 Direct-Push Drilling and Soil Sampling).

8.2 Groundwater Well Installation

The groundwater monitoring wells with be also be installed using DPT. The installation of the groundwater monitoring wells will be performed in general accordance with ADEC's Monitoring Well Guidance, September 2013. The groundwater monitoring wells will be completed with Geoprobe 2.0 in. Slim Prepacks. The prepacked wells will be two inches 2.0-in diameter, prepacked with 20/40 sand, constructed from Schedule 40 PVC with a 5-ft screen section of 0.010 inch slotted screen, riser and threaded end caps. The outer filter pack made from 10/20 sand will be added to 1-ft above the top of the screen, bentonite chips will be added to the near surface. The monitoring wells will be completed with aboveground monuments. No teflon fluids or materials including clothes, pumps, gloves, brushes, soaps and drilling compounds will be used in the installation of the groundwater monitoring wells. Groundwater well installation will be in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling).

8.3 Groundwater Monitoring Well Development, Purging, and Sampling

CES personnel will use a surge block, peristaltic pump and disposable PFAS-free silicone and HDPE tubing to develop the wells. Groundwater monitoring wells will be developed no sooner than 24 hours after installation. The groundwater monitoring wells will be surged and purged in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling).

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After development, at least 48 hours later, CES personnel will use a peristaltic pump and disposable PFAS-free silicone and HDPE tubing to purge the well prior to sampling. Field measurements to include temperature, pH, conductivity, oxygen reduction potential (ORP), and dissolved oxygen (DO) will be collected during purge. When purging monitoring wells prior to sampling, CES will remove at least three casing volumes, monitor water quality parameters until a minimum of three (minimum of four if using temperature as an indicator) of the parameters listed above stabilize, or for low yield wells, the entire well casing is evacuated. The groundwater monitoring wells will be purged and field measurements collected in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling).

After purging, CES personnel will use a peristaltic pump and disposable PFAS-free silicone and HDPE tubing to collect groundwater samples. Prior to handling any groundwater, CES personnel will don a new pair of disposable nitrile gloves which will be interchanged prior to collection of each groundwater sample. Each groundwater sample for PFAS will be collected into two laboratory certified 250 ml-HDPE

containers. Groundwater samples for PFAS will be unpreserved. All containers will be labeled with the project name, sample identification number, date/time of sample collection, preservative, analysis requested, and sampler's initials. The samples will be kept in a sample cooler with double bagged ice (at 4°C ± 2°C) pending delivery to the contract laboratory. The groundwater monitoring wells will be sampled in accordance with CES Standard Operating Procedure SP-02 (SP-02 Groundwater Well Installation, Developing, Purging, and Sampling).

8.4 Equipment and Supplies

Many materials used in environmental investigations can potentially contain PFAS. There is limited published research or guidance on how certain materials used in the field affect sample results. Therefore, a conservative approach will be implemented by CES to exclude materials known to contain PFAS. CES will obtain and review all Material Safety Data Sheets (MSDS) before considering materials for use during PFAS sampling.

CES will avoid the following materials:

- Teflon, polytetrafluoroethylene (PTFE)
- Waterproof coatings containing PFAS
- Food containers
- Anything with fluoro in the name
- Fluorinated ethylene propylene (FEP)
- Ethylene tetrafluoroethylene (ETFE)
- Low density polyethylene (LDPE), polyvinylidene fluoride (PVDF)

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FIELD CLOTHING and PPE

- No clothing or boots containing Gore-Tex®
- All safety boots made from polyurethane and PVC
- No materials containing Tyvek®
- Do not use fabric softener on clothing to be worn in field
- Do not used cosmetics, moisturizers, hand cream, or other related products the morning of sampling
- Do not use unauthorized sunscreen or insect repellent

SAMPLE CONTAINERS

- All sample containers made of HDPE or polypropylene
- Caps are unlined and made of HDPE or polypropylene (no Teflon® -lined caps)

WET WEATHER (AS APPLICABLE)

- Wet weather gear made of polyurethane and PVC only

EQUIPMENT DECONTAMINATION

- PFAS-free water on-site for decontamination of sample equipment
- Only Alconox and Liquinox can be used as decontamination materials

FOOD CONSIDERATIONS

- No food or drink on-site with exception of bottled water and/or hydration drinks (i.e., Gatorade and Powerade) that is available for consumption only in the staging area

FIELD EQUIPMENT

- Must not contain Teflon® (aka PTFE) or LDPE materials
- All sampling materials must be made from stainless steel, HDPE, acetate, silicon, or polypropylene
- No waterproof field books can be used
- No plastic clipboards, binders, or spiral hard cover notebooks can be used
- No adhesives (i.e. Post-It® Notes) can be used
- Sharpies and permanent markers not allowed; regular ball point pens are acceptable
- Aluminum foil must not be used
- Keep PFC samples in separate cooler, away from sampling containers that may contain PFAS
- Coolers filled with regular ice only - Do not use chemical (blue) ice packs

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8.5 Sample Containers and Preservation

Sample container types will be consistent with those specified by Test America, Inc. in Modified Method 537. Tables 1 provides the sample container types, preservative requirements, and hold times for soil and groundwater. Sample containers are those specified in the analytical method, provided by the laboratory selected to perform the analyses, and should be certified by the laboratory to be PFAS-free. The term PFAS-free is a method or project-defined concentration level (for example, < 1/2 the limit of quantitation for the specific compound of interest). Extra containers will be available in case of breakage, contamination, or collection of additional samples.

Best practices in sample preparation must be used when selecting the size, volume, and representativeness of samples. To minimize effects from analyte sorption on sample containers, the laboratory must analyze the entire sample, including the sample container rinsate. The project screening or applicable regulatory

levels, and the expected or potential concentration of the analytes, are also relevant. Because the concentration level of PFAS in aqueous samples determines whether the whole sample or an aliquot is used in the laboratory preparation, CES will collect an additional volume of each sample in a separate container. Then, the laboratory can screen the extra sample for high concentrations without affecting the final sample result.

8.6 Reporting Limits and Method Detection Limits

Reporting limits (RLs) and method detection limits (MDLs) will be consistent with those specified by Test America, Inc. in Modified Method 537. Tables 2 provides the RL and MDLs for soil and groundwater.

8.7 Quality Control Field Sample Collection

Measures of quality include the appropriateness and accuracy of the sample collection; adherence to sample handling protocols; the quality and appropriateness of the laboratory analysis; and the representativeness of the data with respect to the study objectives. Modified Method 537 requires a field reagent blank be collected at each site where field samples are collected. Trip blanks are not applicable to Method 537 and additionally with the use of an isotope dilution method, Modified 537, the inclusion of matrix spike/matrix spike duplicates (MS/MSDs) is unnecessary. Table 3 lists the minimum field QC samples, applicability, and allowable tolerance.

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Field Duplicate Samples

CES will collect a minimum of one field duplicate for every 10 field samples for each matrix sampled and for each target analyte. Field duplicates will be collected from locations of known or suspected contamination, and duplicate soil and water samples will be collected in the same manner and at the same time and location as the primary sample. For sampling occurring over multiple days, all field duplicates will not be collected in one day and the goal will be to collect a minimum of one field duplicate per day. Field duplicates will be submitted as blind samples to the laboratory for analysis, given unique sample numbers (or names) and sample collection time, and adequately documented in the field record or log book. Field duplicate results will be used to calculate and report a precision value for field sampling quality control.

Equipment Blanks

CES will collect a minimum of one equipment blank per 20 samples per matrix to trace sources of artificially introduced contamination. Equipment blanks will consist of water (reagent-grade) supplied by the laboratory poured over or through decontaminated field sampling equipment prior to the collection of environmental samples . Equipment blanks will be given unique sample numbers (or names) and sample

collection time, and adequately documented in the field record or log book. Equipment blanks will not be submitted blind to the laboratory.

Field Blanks (Reagent Blanks)

CES will collect a minimum of one field blank (reagent blank) per 20 samples per matrix to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected. Field blanks (reagent blanks) will consist of water (reagent-grade) supplied by the laboratory, transported to the sampling site, handled like an environmental sample (exposed to sampling equipment/ materials), and returned to the laboratory for analysis. The field blank will be created by attaching 10 feet of new tubing to the peristaltic pump head and withdrawing enough volume of laboratory-supplied water to fill two sample containers or by pouring water over the soil spoons to fill two sample containers. Field blanks (reagent blanks) will be submitted blind to the laboratory.

Temperature Blank

A temperature blank will accompany every cooler transporting samples to the laboratory, the laboratory will measure the temperature of the temperature blank and compare the results against the QA/QC requirement of 4 ± 2 °C during sample receipt procedures.

8.8 Equipment Decontamination Procedures

Field sampling equipment, including oil/water interface meters, direct-push tooling, and other non-dedicated equipment used at each sample location, will be decontaminated between use. CES will use Alconox detergent which is PFAS-free and CBJ public water. CES will use laboratory-certified PFAS-free water for the final rinse during decontamination of non-dedicated sampling equipment (if applicable). Decontamination of direct-push tooling will be with Alconox detergent and CBJ public water. The equipment decontamination will be in accordance with CES Standard Operating Procedure SP-04 (SP-04 Equipment Decontamination Procedures).

8.9 Instrument Calibration Procedures

All field instruments will be calibrated prior to each project according to manufacturer's specifications and instrument calibration must be checked and documented on-site on a daily basis. Certain field screening parameters may require more frequent calibrations depending on site conditions. CES will retain a reference copy of manufacturer's operating instructions in the field. All CES instrument users must be trained in routine maintenance and operation. Calibration standard(s), dates, times and all calibration results will be recorded in the field record or log book. The instrument calibration will be in accordance with CES Standard Operating Procedure SP-04 (SP-03 Instrument Calibration Procedures).

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9 Investigation-Derived Wastes (IDW)

During the site investigation, CES will generate different types of potentially contaminated IDW that include the following:

- Used personal protective equipment (PPE)
- Disposable sampling equipment
- Decontamination fluids
- Soil cuttings from soil borings
- Purged groundwater

Used PPE and disposable sampling equipment will be double bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to the local landfill. Any PPE and disposable equipment that is to be disposed of which can still be reused will be rendered inoperable before disposal in the refuse dumpster.

Decontamination fluids (residual contaminants, water with non-phosphate detergent) and purged groundwater (contaminants, water) will be containerized in 55-gallon, Department of Transportation (DOT) approved, steel drums labeled as to type of waste (water), the source location, and date. Water IDW will then be filtered on-site with a portable granular activated carbon (GAC) filter system and reapplied to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies.

Soil cuttings from soil borings will be containerized in a 55-gallon, DOT approved, steel drum labeled as to type of waste (soil), the source location, and date. Soil IDW will be transported to NRC Alaska for disposal and treatment after an ADEC Soil Transport Form is completed and approved by ADEC.

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10 ADEC Cleanup Levels

On October 1, 2018, ADEC issued proposed regulatory cleanup levels for six PFAS in soil and groundwater for public comment. The comment period closed November 13, 2018. At this time, the proposed amendments are on hold by the department. In April 9, 2019, ADEC published a revised Technical Memorandum on Action Levels for PFAS that supersedes the 2018 action levels memorandum and aligns the action levels with USEPA's Lifetime Health Advisory (LHA) levels for PFOS and PFOA. Action levels serve as thresholds for determining when responsible parties need to provide water treatment or alternative water sources for impacted water supplies.

According to the April 9, 2019 revised Technical Memorandum, in order to align state actions to the recently announced USEPA plans, **ADEC will use the USEPA LHA (PFOS+PFOA above 0.07 µg/L) as the Action Level. Any new testing for PFAS will be for PFOS and PFOA only.**

For the purposes of this investigation, soil analytical data will be compared to the over 40-inch cleanup levels in 18 AAC 75.341 Table B1. Method Two – Soil Cleanup Levels Table. **The PFOS Human Health cleanup level is 1.3 mg/kg and the Migration to Groundwater Cleanup level is 0.0030 mg/kg. The PFOA Human Health cleanup level is 1.3 mg/kg and the Migration to Groundwater Cleanup level is 0.0017 mg/kg.**

Groundwater analytical data will be compared to ADEC groundwater cleanup levels in 18 AAC 75.341 Table C. Groundwater Cleanup Levels. **The PFOS Groundwater Cleanup level is 0.40 µg/L and the PFOA Groundwater Cleanup levels is 0.40 µg/L. Groundwater analytical data will also be compared to the USEPA LHA (PFOS+PFOA above 0.07 µg/L).**

11 Documentation and Reporting

Records shall be maintained as necessary for implementing and documenting the field sampling activities. CES personnel will document all field readings, sample locations, and field observations in a field record or log book. Logbooks or field records must be bound books that are permanently assigned to a specific project. Field forms and camera may also be used for field documentation in a variety of activities. Field forms include borehole logs, well construction, well sampling, site safety and health plan forms, etc. It is not necessary to duplicate information recorded on a field form into the logbook. All logbooks and field form entries must be printed legibly using a waterproof pen. All field forms must be completed in full on a daily basis. Entries to the field notebooks must include the following items if applicable:

- Project name/Site ID/Client/Page Number
- Date
- Weather, site conditions, and other salient observations
- Full name of on-site personnel, affiliations and project title e.g., team leader (including all visitors)
- Daily objectives
- Time and location of activities
- Field observations and comments
- Deviations from the CSP site-specific approved work plan
- Photographic log (photographic name, roll or frame number, description of photograph, date, and time)

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- Site sketches with reference to north direction, sample and field screening locations and depths, and on-site groundwater flow direction
- Survey and location (latitude and longitude coordinates when possible)
- All field measurements (e.g. leak check results, geochemical parameters, field screening results)
- Daily equipment calibrations and maintenance
- Sample record (sample identification, date, time, media, number of samples, and location)
- Cleanup or remediation activities (system performance, system calibration or maintenance record, excavation activities and volume of material removed)
- Waste tracking (when, how much, destination)
- Soil boring logs will include: visual or olfactory observations, field screening readings, soil type, soil moisture, groundwater depth if encountered

CES personnel will correct erroneous field record or log book entries with a single line through the error. Do not erase incorrect information. Date and initial revised entries. Logbooks and field forms will be kept in the project file when complete or when not in use. The documentation will be in accordance with CES Standard Operating Procedure SP-05 (SP-05 Field Documentation).

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The records and any other associated forms will then be used to prepare a report to be submitted to ADEC. This report will provide written documentation of field sampling activities, provide figures showing the sampling locations, and summarize analytical data and resultant comparisons to existing ADEC action levels. This report will be completed after receipt of all analytical data from the contract laboratory.

12 Qualified Person

The field work conducted under this SAP will be conducted by a "Qualified Environmental Professional". Table 4 identifies the project personnel, responsibilities, and contact information for this project.

According to 18 AAC 78.088 an individual is a "Qualified Environmental Professional" if the individual is an impartial third party; is qualified to perform site characterization and cleanup activities, including fate and transport analysis; remediation design; and other activities associated with contaminated sites; actively practices in the field of environmental science or another related scientific field; has not been found to have falsified environmental data or committed other acts of fraud directly related to environmental work; and meets one or more of the following minimum educational qualification and experience requirements: has a four-year undergraduate or a graduate degree from a nationally or internationally accredited post-secondary institution in environmental science or another related scientific

field, and has at least one year of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional completed after the degree described in this sub-paragraph was obtained; has a four-year degree from a nationally or internationally accredited post-secondary institution in any field or a two-year associate degree from a nationally or internationally accredited post-secondary institution in environmental science or another related scientific field, and has at least three years of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional completed after a degree described in this sub-paragraph was obtained; is certified as an environmental technician under an apprenticeship program with a registration under 29 C.F.R. Part 29, and has at least three years of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional completed after the certification described in this sub-paragraph was obtained.

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Title 14 Code of Federal Regulation (CFR) Part 139

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ATTACHMENT A

TABLES & FIGURES

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Table 1. Sample container types, preservative requirements, and hold times for PFAS samples.

Analysis	Method Number	Container	Preservative	Maximum Holding Times
PFAS - Soil	Modified Method 537	1 – 4 oz HDPE jar	0°C to 6°C	14 days
PFAS - Groundwater	Modified Method 537	2- 250 ml HDPE containers	0°C to 6°C	14 days

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Table 2. Reporting Limits and Method Detection Limits for PFAS samples.

Analysis	Constituent	RL	MDL	Units
PFAS Soil – Method 537 Modified	PFBS	0.200	0.0250	µg/kg
	PFHxS	0.200	0.0310	µg/kg
	PFHpA	0.200	0.0290	µg/kg
	PFOA	0.200	0.0860	µg/kg
	PFOS	0.500	0.200	µg/kg
	PFNA	0.200	0.0360	µg/kg
Isotope Dilution 18O2 PFHxS, 13C4 PFHpA, 13C4 PFOA, 13C4 PFOS, 13C3 PFBS, 13C5 PFNA				
PFAS Groundwater – Method 537 Modified	PFBS	2.00	0.200	ng/L
	PFHxS	2.00	0.170	ng/L
	PFHpA	2.00	0.250	ng/L
	PFOA	2.00	0.850	ng/L
	PFOS	2.00	0.540	ng/L
	PFNA	2.00	0.270	ng/L
Isotope Dilution 18O2 PFHxS, 13C4 PFHpA, 13C4 PFOA, 13C4 PFOS, 13C3 PFBS, 13C5 PFNA				

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Table 3. Minimum Quality Control Requirements.

Minimum Field QC Samples	Applicability	Allowable Tolerance
Field Duplicate (Minimum of one per every 10 field samples for each matrix sampled, for each day in field, for each target analyte, minimum of one)	All soil & water samples collected on the same day	Relative percent differences (RPD) less than: 30% water, 50% soil
Decontamination or Equipment Blank (One per set of 20 similar samples, minimum of one)	Per project specifications, to include soil sampling spoons, groundwater sampling tubing, and/or direct push tooling.	Less than the practical quantitation limit
Field Reagent Blank (One per set of 20, minimum of one)	Per project specifications and per Modified Method 537	Less than the practical quantitation limit
Temperature Blank	Per project specifications, one per cooler	4°C+/-2°C

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Table 4. Key project personnel and contact information.

Key Project Position	Contact Information
CES Project Manager (PM)/ Qualified Environmental Professional	Jolene M Cox, Principal Environmental Scientist Cox Environmental Services 712 W 12th Street, Juneau, AK 99801 Telephone: 907-586-4447 (office) 907-723-9946 (cell) e-mail jcox@coxenv.com
Laboratory Project Manager	Sheri Cruz, Project Manager Test America, Inc. 5755 8th Street East Tacoma, WA 98424 Telephone: 253-248-4960 (office) e-mail sheri.cruz@testamericainc.com
CES Project Chemist/Data Validation	Jolene M Cox
ADEC Project Manager (PM)	Danielle Duncan Alaska Department of Environmental Conservation Division of Spill Response Contaminated Sites Program P.O. Box 111800, Juneau AK 99811 Telephone: 907-465-5207 (office) e-mail danielle.duncan@alaska.gov
Emergency – Police Department, Fire Department, Ambulance	911
Emergency Medical Facility	Bartlett Regional Hospital 3260 Hospital Dr, Juneau, AK 99801 Telephone: 907-796-8900
Poison Control Center	Telephone: 1-800-222-1212

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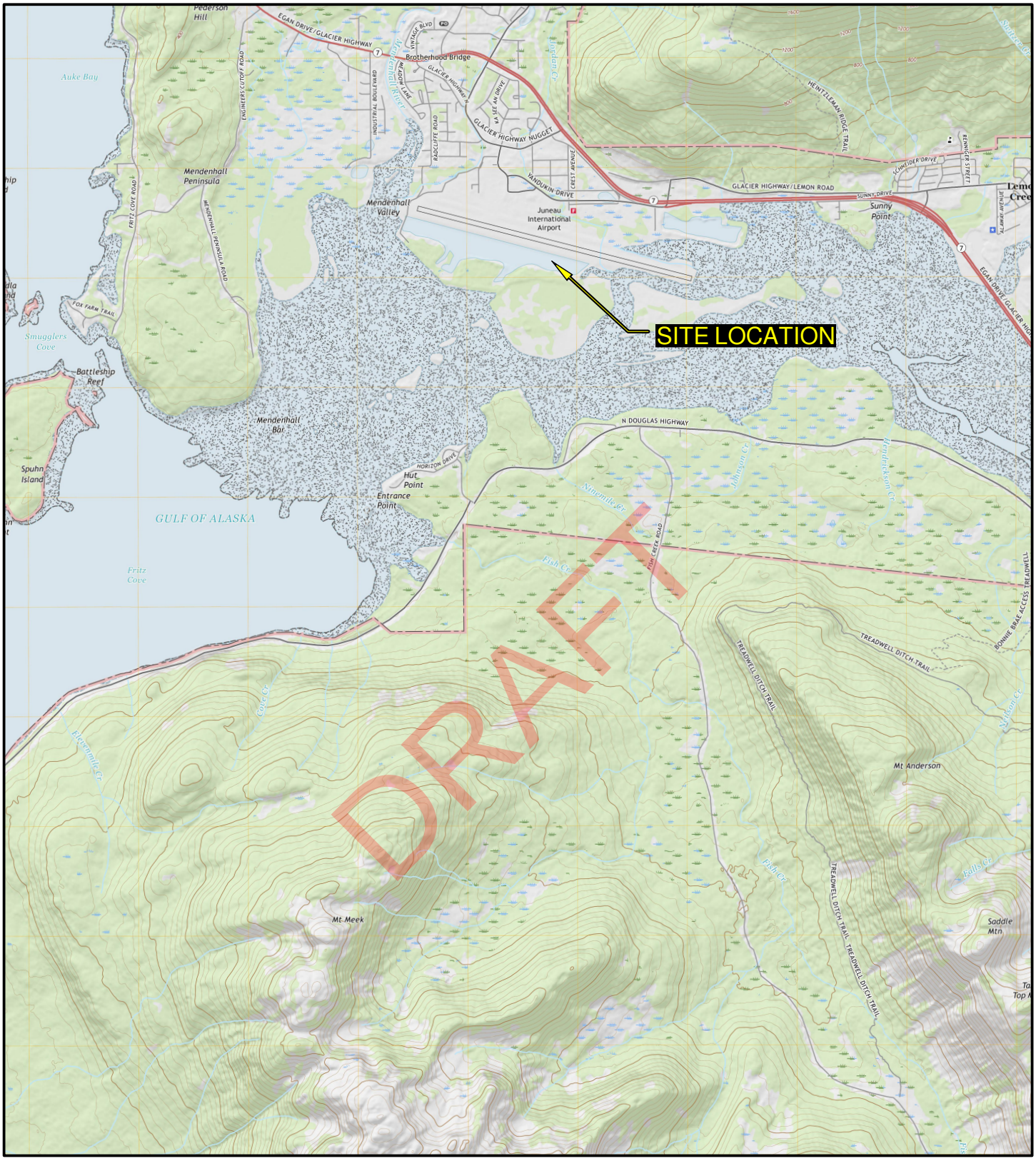


COX environmental services

712 W 12th Street Juneau, Alaska 99801 907.586.4447 www.coxenv.com

Prepared for: City & Borough of Juneau

FIGURE 1. SITE LOCATION MAP

Juneau International Airport
1873 Shell Simons Drive
Juneau, AK

Date:7.18.2019

COX environmental services
712 W 12th Street Juneau, Alaska 99801 907.586.4447 www.coxenv.com

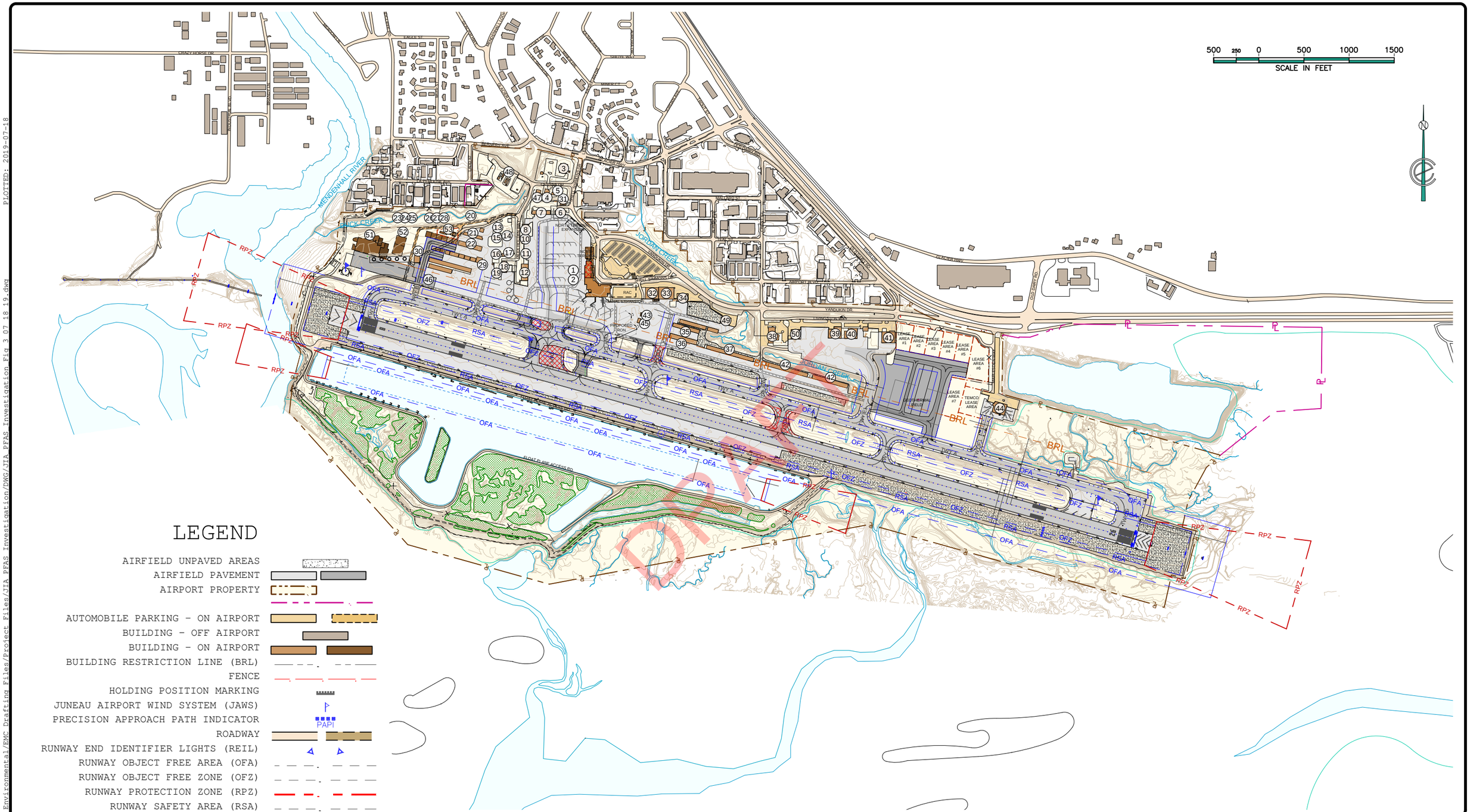
2013 Aerial Photograph

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE
DRAWN emc
CHECKED jmc
DATE 7/18/2019

Fig 2

FILE: /home/iolo/Cox Environmental/EMC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 2 07 18 19.dwg PLOTTED: 2019-07-18



LEGEND

- AIRFIELD UNPAVED AREAS
- AIRFIELD PAVEMENT
- AIRPORT PROPERTY
- AUTOMOBILE PARKING – ON AIRPORT
- BUILDING – OFF AIRPORT
- BUILDING – ON AIRPORT
- BUILDING RESTRICTION LINE (BRL)
- FENCE
- HOLDING POSITION MARKING
- JUNEAU AIRPORT WIND SYSTEM (JAWS)
- PRECISION APPROACH PATH INDICATOR (PAPI)
- ROADWAY
- RUNWAY END IDENTIFIER LIGHTS (REIL)
- RUNWAY OBJECT FREE AREA (OFA)
- RUNWAY OBJECT FREE ZONE (OFZ)
- RUNWAY PROTECTION ZONE (RPZ)
- RUNWAY SAFETY AREA (RSA)
- TAXIWAY OBJECT FREE AREA (TOFA)
- TO BE REMOVED
- TOPOGRAPHIC CONTOUR
- TREES
- VISUAL APPROACH SLOPE INDICATOR (VASI)
- WIND SOCK

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Site Plan

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE 1"=1000'
DRAWN jmc
CHECKED jmc
DATE 7/18/2019

Fig 3

FILE: /home/dojo/Cox Environmental/ENC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 3 07 18 19.dwg
PLOTTED: 2019-07-18

#	DESCRIPTION
1	TERMINAL BUILDING
2	AIRPORT TRAFFIC CONTROL TOWER (ATCT)
3	AFFSS/SACOM
4	J/D TELEPHONE
5	LEASED TO HERTZ
6	AERO SERVICES (SUBLEASE: FEDEX) R&L LEASING
7	AIRFIELD MAINTENANCE FACILITY
8	LAB FLYING
10	HEIMBIGNER
11	ALASKA AIRLINES
12	ALASKA AIRLINES
13	PRIVATE HANGAR
14	FISH AND WILDLIFE
15	3 PRIVATE T-HANGARS
16	DUANE PACKER (SUBLEASE: UNITED PARCEL SERVICE)
17	R&L LEASING
18	AERO SERVICES
19	AERO SERVICES
20	PRIVATE HANGAR
21	T-HANGAR (7 UNITS)
22	T-HANGAR (12 UNITS)
23	PRIVATE HANGAR
24	PRIVATE HANGAR
25	PRIVATE HANGAR
26	PRIVATE HANGAR
27	PRIVATE HANGAR
28	PRIVATE HANGAR
29	PRIVATE HANGAR
30	CIVIL AIR PATROL
31	DELTA WESTERN
32	SAND STORAGE [POTENTIAL ATCT LOCATION]
33	LOKEN AVIATION
34	WARD AIR (RED LEASING)
35	BLOCK 'L' EXECUTIVE HANGARS (5 UNITS)
36	BLOCK 'M' T-HANGARS (14 UNITS)
37	BLOCK 'N' T-HANGARS (9 UNITS)
38	AIRCRAFT RESCUE AND FIRE FIGHTING (ARFF)
39	SILVER BAY LOGGING
40	ALASKA NATIONAL GUARD
41	WINGS OF ALASKA
42	BLOCK 'O' EXECUTIVE HANGARS (14 UNITS)
43	COASTAL FUEL
44	TEMSCO HELICOPTERS
45	COASTAL HELICOPTERS
46	BLOCK 'I' HANGARS
47	R&L LEASING
48	FUEL FARM
49	ALASKA SEAPLANES HANGAR
50	GLACIER AVIATION (AIRLIFT NORTHWEST) HANGAR
51	FUTURE SNOW REMOVAL EQUIPMENT BUILDING (SREB)
52	FUTURE SAND CHEMICAL BUILDING
53	FUTURE FUEL FACILITY

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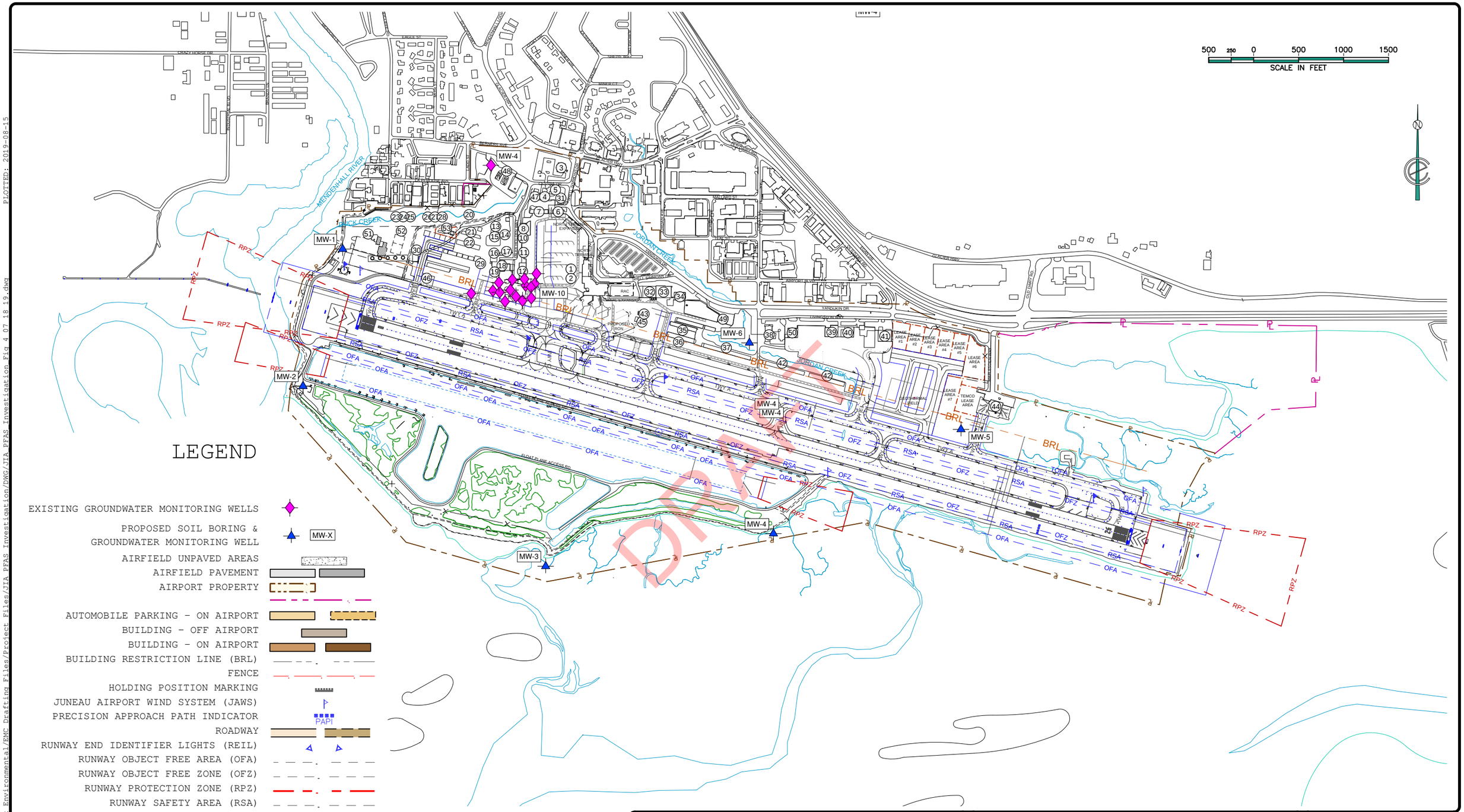
AIRPORT FACILITIES

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA



LEGEND

- EXISTING GROUNDWATER MONITORING WELLS
- PROPOSED SOIL BORING & GROUNDWATER MONITORING WELL (MW-X)
- AIRFIELD UNPAVED AREAS
- AIRFIELD PAVEMENT
- AIRPORT PROPERTY
- AUTOMOBILE PARKING – ON AIRPORT
- BUILDING – OFF AIRPORT
- BUILDING – ON AIRPORT
- BUILDING RESTRICTION LINE (BRL)
- FENCE
- HOLDING POSITION MARKING
- JUNEAU AIRPORT WIND SYSTEM (JAWS)
- PRECISION APPROACH PATH INDICATOR (PAPI)
- ROADWAY
- RUNWAY END IDENTIFIER LIGHTS (REIL)
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- RUNWAY OBJECT FREE ZONE (OFZ)
- RUNWAY PROTECTION ZONE (RPZ)
- RUNWAY SAFETY AREA (RSA)
- TAXIWAY OBJECT FREE AREA (TOFA)
- TO BE REMOVED
- TOPOGRAPHIC CONTOUR
- TREES
- VISUAL APPROACH SLOPE INDICATOR (VASI)
- WIND SOCK

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712 W 12th Street Juneau, Alaska 99801 907.586.4447 www.coxenv.com

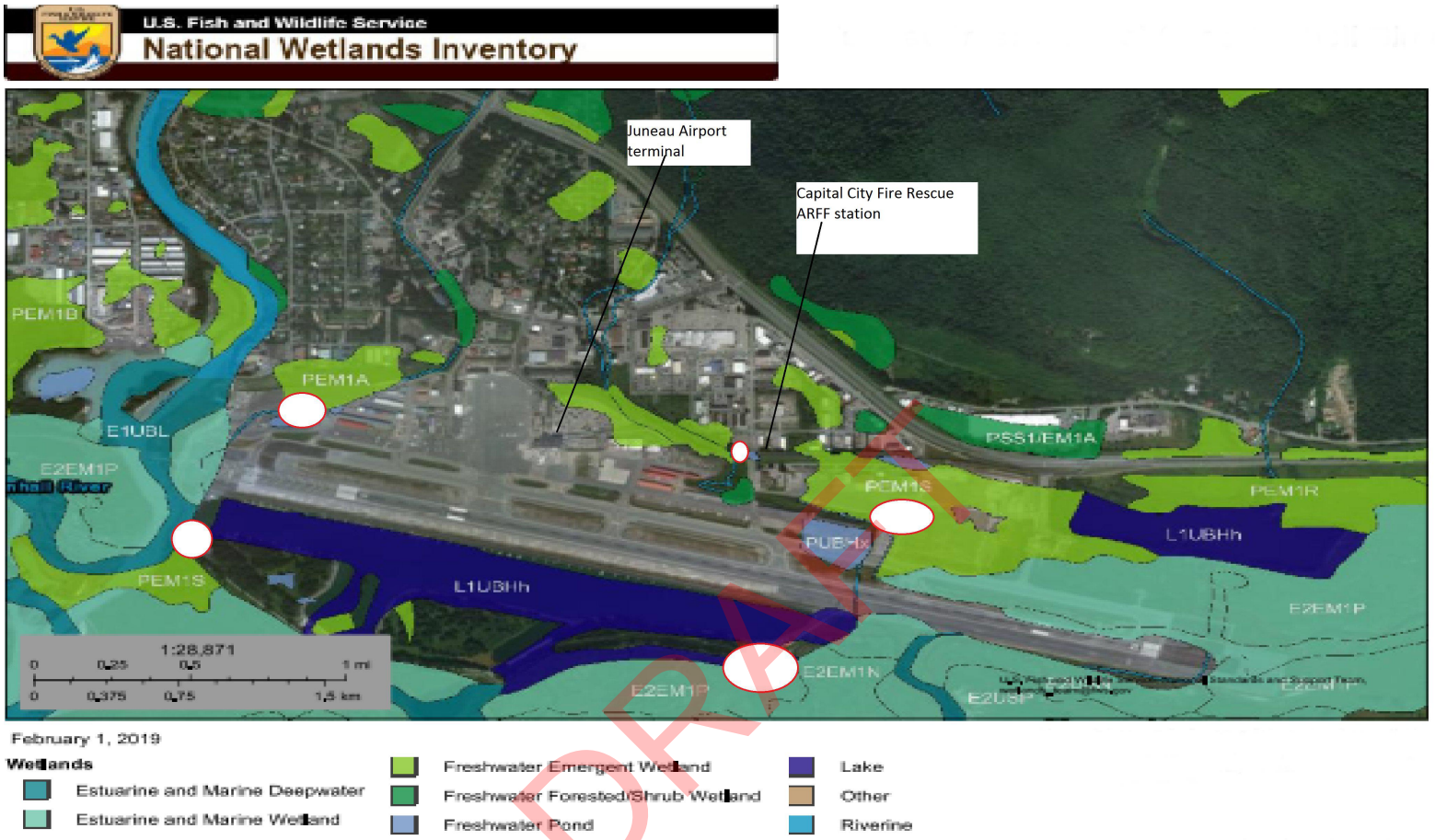
Proposed Soil Borings and Groundwater Monitoring Wells

JUNEAU INTERNATIONAL AIRPORT
JUNEAU, ALASKA

SCALE 1"=1000'
DRAWN jmc
CHECKED jmc
DATE 8/15/2019

Fig 4

FILE: /home/jo1o/Cox Environmental/ENC Drafting Files/Project Files/JIA PFAS Investigation/DWG/JIA PFAS Investigation Fig 4 07 18 19.dwg
PLOTTED: 2019-08-15



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Areas outlined in red are potential places where foam was dispensed.

Image with locations provided by CCFR (2019)

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Prepared for: **City & Borough of Juneau**

FIGURE 5. POTENTIAL AFFF DISCHARGE AREAS

Juneau International Airport
1873 Shell Simmons Drive
____

Date:8.15.2019

ATTACHMENT B

CONCEPTUAL SITE MODEL

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Print Form

HUMAN HEALTH CONCEPTUAL SITE MODEL GRAPHIC FORM

Site: Juneau International Airport
1873 Shell Simons Drive Juneau AK 99801

Completed By: Jolene Cox - Cox Environmental Services
Date Completed: 07 17 19

***Instructions*: Follow the numbered directions below. Do not consider contaminant concentrations or engineering/land use controls when describing pathways.**

(1)
Check the media that could be directly affected by the release.

(2)
For each medium identified in (1), follow the top arrow and check possible transport mechanisms. Check additional media under (1) if the media acts as a secondary source.

(3)
Check all exposure media identified in (2).

(4)
*Check all pathways that could be complete. The pathways identified in this column **must** agree with Sections 2 and 3 of the Human Health CSM Scoping Form.*

(5)
Identify the receptors potentially affected by each exposure pathway: Enter "C" for current receptors, "F" for future receptors, "C/F" for both current and future receptors, or "I" for insignificant exposure.

Current & Future Receptors

Media	Transport Mechanisms	Exposure Media	Exposure Pathway/Route	Residents (adults or children)	Commercial or industrial workers	Site visitors, trespassers, or recreational users	Construction workers	Farmers or subsistence harvesters	Subsistence consumers	Other
☑ Surface Soil (0-2 ft bgs)	☑ Direct release to surface soil — check soil									
	☑ Migration to subsurface — check soil									
	☑ Migration to groundwater — check groundwater									
	☐ Volatilization — check air									
	☑ Runoff or erosion — check surface water									
	☑ Uptake by plants or animals — check biota									
	☐ Other (list):									
☑ Subsurface Soil (2-15 ft bgs)	☑ Direct release to subsurface soil — check soil									
	☑ Migration to groundwater — check groundwater									
	☐ Volatilization — check air									
	☑ Uptake by plants or animals — check biota									
	☐ Other (list):									
☑ Ground-water	☑ Direct release to groundwater — check groundwater									
	☐ Volatilization — check air									
	☑ Flow to surface water body — check surface water									
	☑ Flow to sediment — check sediment									
	☑ Uptake by plants or animals — check biota									
	☐ Other (list):									
☑ Surface Water	☑ Direct release to surface water — check surface water									
	☐ Volatilization — check air									
	☑ Sedimentation — check sediment									
	☑ Uptake by plants or animals — check biota									
	☐ Other (list):									
☑ Sediment	☑ Direct release to sediment — check sediment									
	☑ Resuspension, runoff, or erosion — check surface water									
	☑ Uptake by plants or animals — check biota									
	☐ Other (list):									
		☑ soil	☑ Incidental Soil Ingestion	F	C	I	C			
			☑ Dermal Absorption of Contaminants from Soil	F	C	I	C			
			☐ Inhalation of Fugitive Dust							
		☑ groundwater	☑ Ingestion of Groundwater	F	F	F	F			
			☑ Dermal Absorption of Contaminants in Groundwater	F	F	F	F			
			☐ Inhalation of Volatile Compounds in Tap Water							
		☐ air	☐ Inhalation of Outdoor Air							
			☐ Inhalation of Indoor Air							
			☐ Inhalation of Fugitive Dust							
		☑ surface water	☑ Ingestion of Surface Water	F	F	F	F			
			☑ Dermal Absorption of Contaminants in Surface Water	F	F	F	F			
			☐ Inhalation of Volatile Compounds in Tap Water							
		☑ sediment	☑ Direct Contact with Sediment	F	C	I	C	C/F	C/F	
		☑ biota	☑ Ingestion of Wild or Farmed Foods	F				C/F	C/F	

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Revised, 10/01/2010

ATTACHMENT C

COX ENVIRONMENTAL SERVICES STANDARD OPERATING PROCEDURES (SOPs)

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cox environmental

CES-SOP-01: Direct-Push Soil Sampling

All direct-push soil sampling will be conducted per the following Alaska Department of Environmental Conservation (ADEC) guidance documents:

- **Field Sampling Guidance (ADEC, 2017)**
- **Underground Storage Tanks Procedures Manual: Guidance for Treatment of Petroleum-Contaminated Soil and Groundwater and Standard Sampling Procedures (ADEC, 2017)**

Direct Push Drilling

Direct push system (DPS) soil samples will be collected using Geoprobe's® Macrocore M5 Sampling System sampler with polyvinyl chloride (PVC) liners.

1. At the top of each sample interval, the continuous tube sampler will be driven into the substrate to a depth equal to the length of the sampler.
2. After the sampler has been advanced, it is retrieved from the borehole and the PVC liner containing the soil core is placed on a firm, horizontal surface, for opening, inspection, and sampling.
3. The PVC liner for each sample core is then cut open to expose the soil sample core for soil sampling and examination.
4. Samples for laboratory analysis will be immediately transferred into clean laboratory sample containers using a disposable polyethylene spoon or scoops.
5. The soil core will then be examined, screened for VOCs using a PID, and logged for lithology if necessary, CES will log soils after sample collection). The soil cores will also be photographed.
6. The cores will be labeled so that the up/down orientation is not lost.

Soil Sampling Procedure

The following procedures will be used to collect soil samples for laboratory analysis.

1. Soil samples will be collected with disposable or clean tools that have been decontaminated.
2. Disposable nitrile gloves will be worn and changed between sample collections.
3. Soil samples will be placed in containers quickly and in the order of volatility.
4. Soil samples collected for analyses other than VOCs, GRO, or VPH will be thoroughly homogenized using a polyethylene sampling spoon or Ziploc® (or similar) re-sealable bags.
5. The homogenized material will then be divided equally among the appropriate sample containers.
6. Sample containers will be quickly and adequately sealed, and rims cleaned before tightening lids.
7. Record the sample ID, date and time, sampler, analytes, and other sample information on the sample container labels, the sample chain-of-custody, and field log book.
8. Samples will be preserved immediately according to the method specifications appropriate for the laboratory parameters to be analyzed.
9. Unless specified otherwise, at a minimum, the samples must be immediately chilled to 4 ±2 degrees Celsius (°C) and this temperature must be maintained through delivery to the laboratory for analysis.
10. Preserve containers immediately if preservative is not already in the containers, and unless specified otherwise, at a minimum, immediately cool the samples to 4 ±2°C and maintain this temperature through delivery to laboratory until the samples are analyzed.

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Quality Assurance/Quality Control Samples Procedure
Quality Assurance/Quality Control (QA/QC) samples should be collected during soil sampling according to the site-specific SAP.

Field Duplicate Samples
Field duplicate samples will be collected simultaneously or in immediate succession to the normal samples using identical sampling techniques. Duplicate sample results are used to assess precision of the sample collection process. Duplicate samples will be collected at a frequency of one per ten field samples per matrix sampled. Field duplicates will be submitted blind to the laboratory.

Equipment Blanks
Equipment blanks will consist of water (reagent-grade) supplied by the laboratory, transported to the sampling site, and used to rinse the Geoprobe drive point immediately after decontamination. The equipment blank is created by pouring the reagent-grade water over the drive point and allowing the water to run into the poly sample bottle. Two sample bottles will be filled. One equipment blank will be produced as part of this field effort following the completion of drilling at the first sampling location. Equipment blanks will not be submitted blind to the laboratory.

Matrix Spikes and Matrix Spike Duplicates
Matrix spike (MS) and MS duplicate (MSD) samples are similar to primary samples but provide a quantity of material three times larger to enable the evaluation of matrix effects by the analytical laboratory. Conventional naming is used, but MS/MSD is noted in the analysis field on the CoC form. MS/MSD samples will be designated for each sample shipment/lab batch, or at a frequency of one pair per 20 or fewer primary samples per matrix (5 percent).

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Field Blank (Reagent Blank)
The field blank (reagent blank) will consist of water (reagent-grade) supplied by the laboratory, transported to the sampling site, handled like an environmental sample (exposed to sampling equipment/materials), and returned to the laboratory for analysis. The field blank will be created by attaching 10 feet of new tubing to the peristaltic pump head and withdrawing enough volume of laboratory-supplied water to fill two sample containers. One field blank will be produced as part of this effort. Field blanks (reagent blanks) will be submitted blind to the laboratory.

Temperature Blank
A temperature blank is a high-density polyethylene bottle filled with at least 500 milliliters of water that will accompany every cooler transporting project samples to the laboratory. Upon receiving the sample shipment, the laboratory will measure the temperature of the blank and compare the results against the QA/QC requirement of 4 ± 2 °C during sample receipt procedures and will log the data on the COC.

Geoprobe® Macro-Core® MC5
1.25-inch Light-Weight Center Rod Soil Sampling System

Standard Operating Procedure

Technical Bulletin No. MK3139

PREPARED: January, 2011

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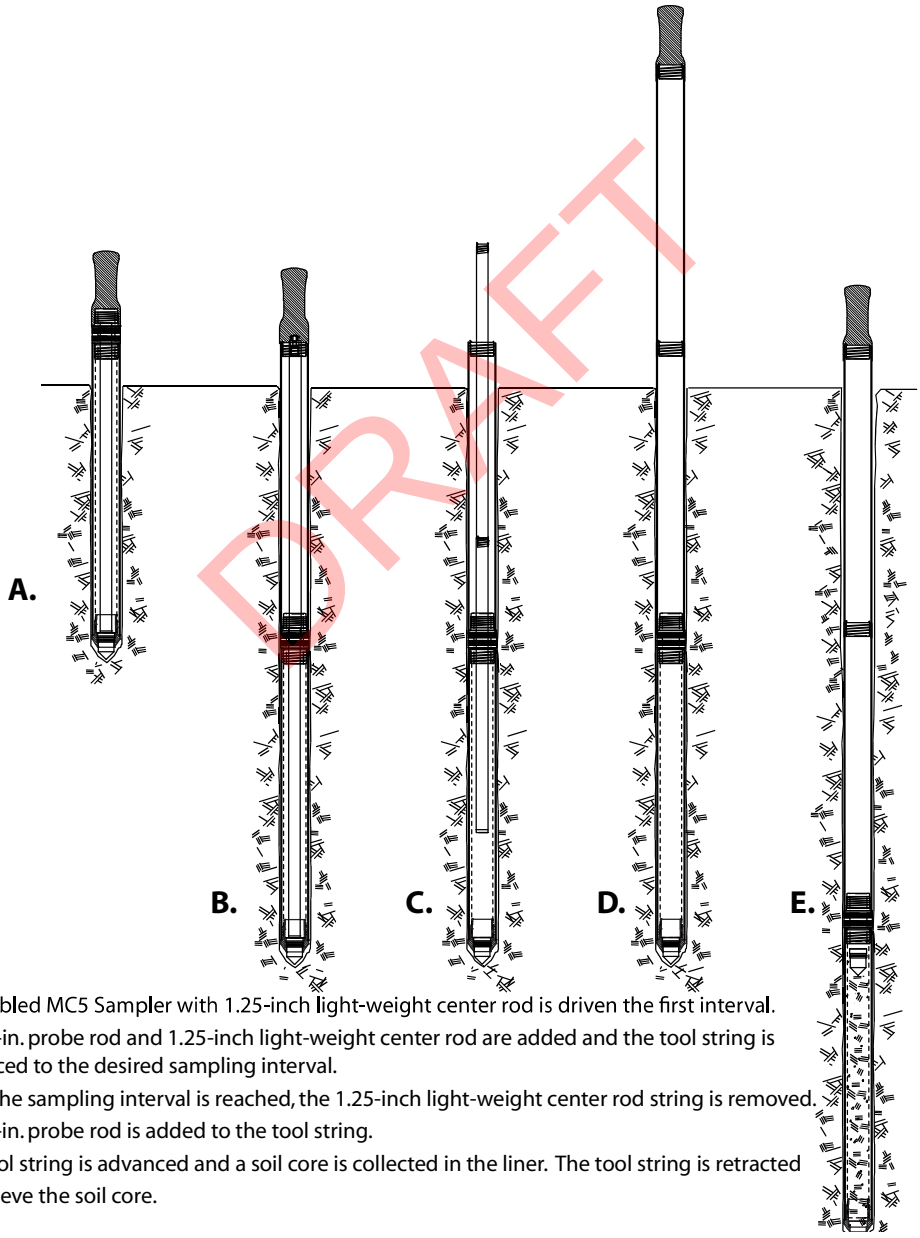

A. Assembled MC5 Sampler with 1.25-inch light-weight center rod is driven the first interval.
B. A 2.25-in. probe rod and 1.25-inch light-weight center rod are added and the tool string is advanced to the desired sampling interval.
C. Once the sampling interval is reached, the 1.25-inch light-weight center rod string is removed.
D. A 2.25-in. probe rod is added to the tool string.
E. The tool string is advanced and a soil core is collected in the liner. The tool string is retracted to retrieve the soil core.

OPERATION OF THE MACRO-CORE® MC5 SOIL SAMPLING SYSTEM

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Macro-Core® and Large Bore Soil Samplers manufactured under US Patent 5,606,139.

Macro-Core® Closed-Piston Drive Point manufactured under US Patent 5,542,481

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1.0 OBJECTIVE

The objective of this procedure is to collect a representative soil sample at depth and recover it for visual inspection and/or chemical analysis.

2.0 BACKGROUND

2.1 Definitions

Geoprobe®*: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.

**Geoprobe® and Geoprobe Systems® are registered trademarks of Kejr, Inc., Salina, Kansas.*

Macro-Core® MC5 Soil Sampler:** A solid barrel, direct push device for collecting continuous core samples of unconsolidated materials at depth. Although other lengths are available, the standard Macro-Core® MC5 Sample Tubes come in lengths of 48 inches and 60 inches with an outside diameter of 2.25 inches. Samples are collected inside a removable liner. The Macro-Core® MC5 Sampler may be used in an open-tube or closed-point configuration.

***Macro-Core® is a registered trademarks of Kejr, Inc., Salina, Kansas.*

Liner: A removable/replaceable, thin-walled tube inserted inside the Macro-Core® MC5 sample tube for the purpose of containing and storing soil samples. While other lengths are available, the most common Macro-Core® MC5 Liners are 48 inches and 60 inches in length. The liner length should correspond to the length of the sample tube used. Liner materials include stainless steel, Teflon®, and PVC.

1.25-inch Light-Weight Center Rods: Used as the inner Rod String for Macro-Core® MC5 sampling. 1.25-inch Light-Weight Center rods come in lengths of 48 inches and 60 inches. They provide a weight reduction of up to 64% over standard 1.25-inch probe rods.

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2.2 Discussion

In this procedure, an assembled Macro-Core® MC5 Soil Sampler is driven one sampling interval into the subsurface and retrieved using a Geoprobe® direct push machine. The collected soil core is removed from the sampler along with the used liner. After decon, the Macro-Core® sampler is reassembled using a new liner. The clean sampler is then advanced back down the same hole to collect the next soil core. The Macro-Core® Sampler may be used as an open-tube or closed-point sampler.

The Macro-Core® MC5 Soil Sampler is commonly used as an open-tube sampler (Fig. 2.1A). In this configuration, coring starts at the ground surface with a sampler that is open at the leading end. The sampler is driven into the subsurface and then pulled from the ground to retrieve the first soil core. In stable soils, an open-tube sampler is advanced back down the same hold to collect the next core.

In unstable soils which tend to collapse into the core hold, the Macro-Core® MC5 Sampler can be equipped with a 1.25-inch Center Rod Closed-Point assembly (Fig 2.1B). The point fits firmly into the cutting shoe and is held in place by the 1.25-inch light-weight center rods. The Macro-Core® MC5 Center Rod System prevents collapsed soil from entering the sampler as it is advanced to the bottom of an existing hole, thus ensuring collection of a representative sample. Once the 1.25-inch light weight center rod system is removed, the point

will be pushed up the liner during the next sampling interval. The point assembly is later retrieved from the sampler with the liner and soil core.

The Macro-Core® MC5 Soil Sampler is a true discrete sampler. It can be driven through undisturbed soil to a desired depth using the 1.25-inch Light Weight Center Rod System. Once the 1.25-inch light-weight center rods are removed, a representative sample is recovered from the desired depth.

Loose soils may fall from the bottom of the sampler as it is retrieved from depth. The MC Core Catcher (Fig. 3.1) alleviates this problem. Excellent results are obtained when the core catcher is used with saturated sands and other non-cohesive soils. A core catcher should not be used with tight soils as it may actually inhibit sample recovery. In that case, a MC Spacer Ring or extended shank cutting shoe can be used. Constructed of PVC, the core catcher is suitable for use with all Geoprobe® liners.

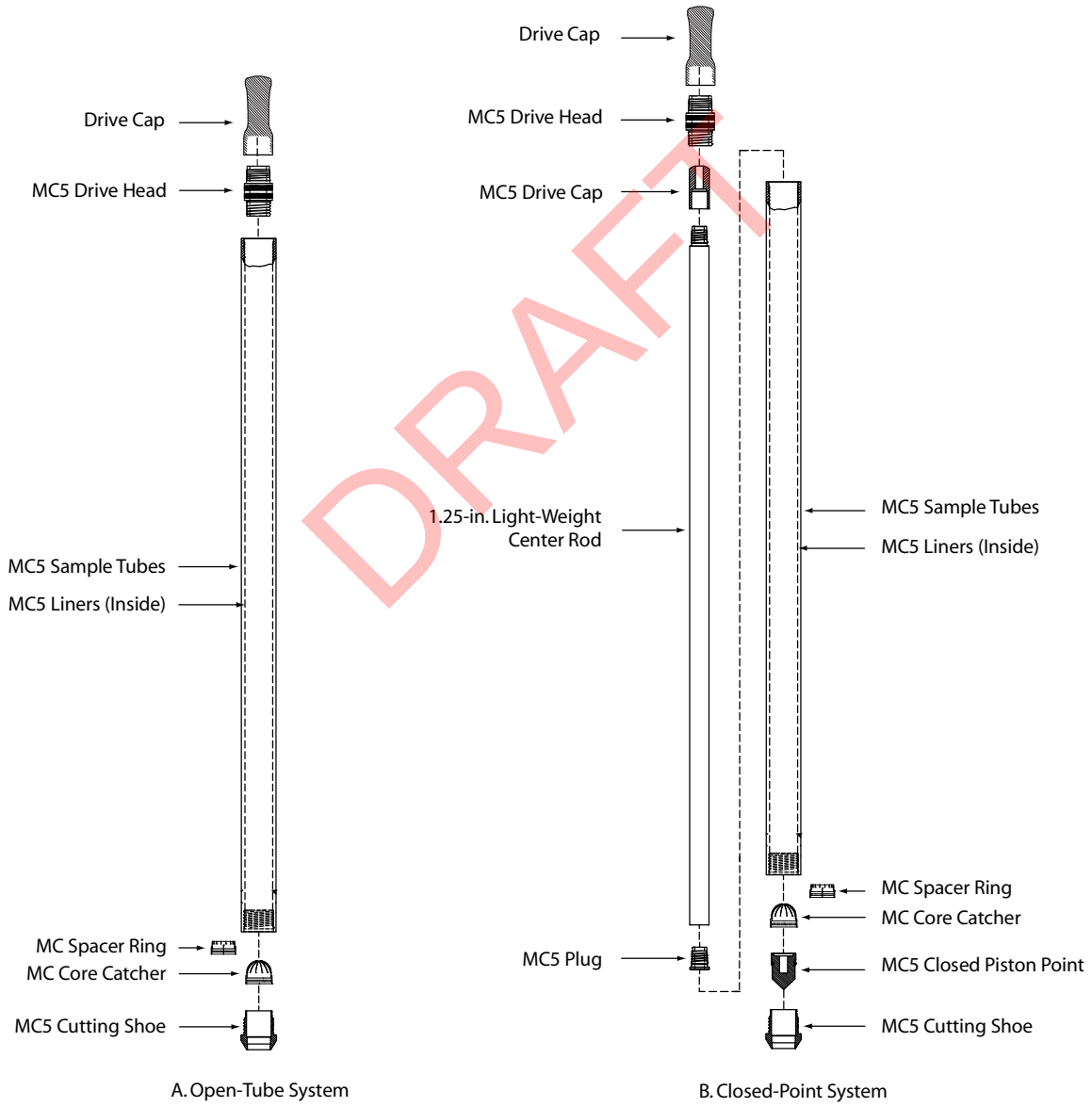

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Figure 2.1
Macro-Core® MC5 Soil Sampler Configurations

3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to recover representative soil cores with the MC5 Soil Sampling System. Sample tubes, 1.25-inch light-weight center rods, probe rods, and liners all need to be of equal length in order to obtain a sample. Refer to Figure 3.1 for identification of the specified parts. Additional tooling options are available in Appendix A.

MC5 Sampler Parts	Part Number
MC5 Drive Head, 2.25 in. bored	28646
MC5 Drive Head, 2.125 in. bored	23640
MC5 Sample Tubes, 60 in.	22992
MC5 Sample Tubes, 48 in.	22923
MC5 Sample Tubes, 1 m.	24239
MC5 Sample Tubes, 36 in.	24238
MC5 Sample Tubes, 24 in.	24237
MC5 Cutting Shoe, standard, 2.25 in. OD	22922
MC5 Cutting Shoe, undersized, 1.35 in. ID	23957
MC5 Cutting Shoe, standard, 2.25 in. OD (extended shank)	23978
MC5 Cutting Shoe, undersized, 1.35 in. ID (extended shank)	28237
MC5 Cutting Shoe, undersized, 1.25 in. ID (extended shank)	26078
MC5 Cutting Shoe, Heavy Duty, 1.35 in. ID,	29552
MC5 Closed Piston Point, standard	28113
MC5 Closed Piston Point, undersized	26865

Center Rods (1.25 in.) and Center Rod Accessories	Part Number
1.25-in. Center Rod, 60 in. Lightweight	27600
1.25-in. Center Rod, 48 in. Lightweight	21900
Probe Rod, 1.25 in. x 1 m.	AT1239
Probe Rod, 1.25 in. x 36 in.	AT1236
Probe Rod, 1.25 in. x 24 in.	AT1224
MC5 Drive Cap, 1.25 in. Center Rod, Threadless	23639
MC5 Plug Threaded, 1.25 in.	23641
1.25 in. Pull Cap	AT1204

	Part Numbers for Specific Probe Rod OD	
Probe Rods and Probe Rod Accessories	2.25-in. OD	2.125-in. OD
Probe Rod, 60 in.	25301	AT2160
Probe Rod, 48 in.	25300	AT2148
Probe Rod, 1 m.	25352	AT2139
Probe Rod, 2.125 in. x 36 in.		AT2136
Probe Rod, 2.125 in. x 24 in.		13072
Drive Cap, GH60 Series, Threadless.	31530	8397
Drive Cap, GH40 Series, Threadless	31405	
Drive Cap, GH40 Series, Threaded		AT2101
Pull Cap.	25298	AT2104

MC5 Liners, Accessories, and Miscellaneous Tools	Part Number
MC Liners, 60 in. (66 liners)	10074
MC Liners, 48 in. (66 liners)	AT927K
MC Liners, 1m. (66 liners)	AT928K
MC Liners, 36 in. (66 liners)	AT921K
MC Liners, 24 in. (66 liners)	AT926K
MC Core Catcher	AT8531
MC Spacer Ring	AT8532
MC Spacer Ring (Bulk Box of 500)	AT8533K
Vinyl End Caps (Package of 66)	AT726K
Liner Cutter	AT8010
Universal Liner Holder	22734
Rod Wiper Weldment	23633
Rod Wiper Doughnuts, 2.125-in and 2.25-in.	26876
Two Pipe Wrenches	

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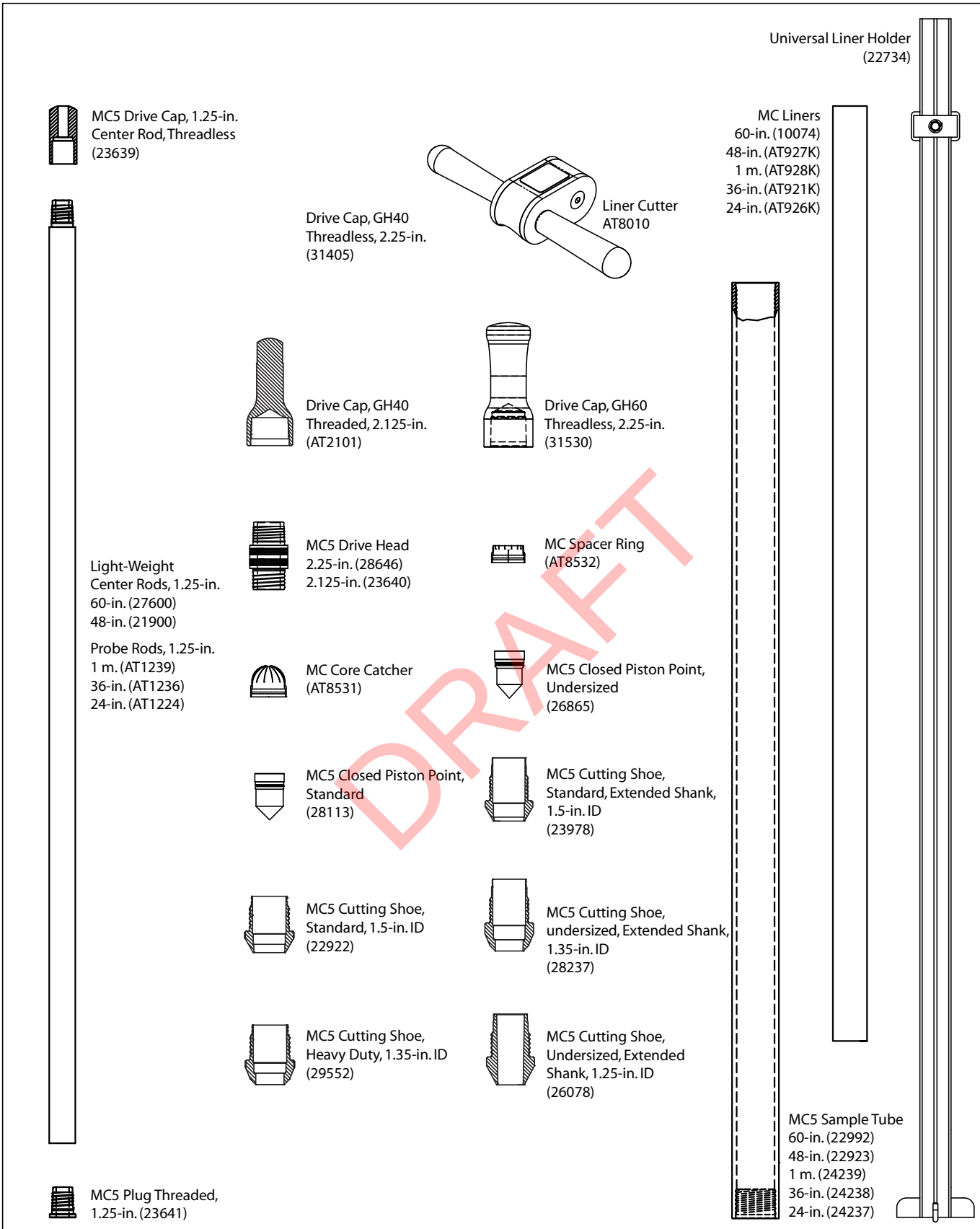

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Figure 3.1
Macro-Core® MC5 Soil Sampler Parts

3.1 Tool Options

Five major components of the MC5 Soil Sampling System are sample tubes, probe rods, 1.25-inch light-weight center rods, sample liners, and cutting shoes. These items are manufactured in a variety of sizes to fit the specific needs of the operator. This section identifies the specific tool options available for use with the MC5 Soil Sampling System.

Sample Tubes
MC5 Sample tubes come in lengths of 60 inches (1524 mm), 48 inches (1219 mm), 1 meter, 36 inches (914 mm), and 24 inches (610 mm).

Probe Rods
Standard Geoprobe® 2.125-inch and 2.25-inch OD probe rods are required to operate the MC5 Soil Sampling System. The specific length of rods may be selected by the operator. The most common rod lengths used in MC5 Soil Sampling are the 60-inch and 48-inch rods.

1.25-inch Light-Weight Center Rods
1.25-inch Light-Weight Center Rods (1.25-inch / 32-mm OD) are recommended for the inner rod string of the MC5 system when utilizing an outer casing of 48- or 60-inch long rods. Choose the light-weight rod length that matches the length of rods used for the outer casing (48-inch light-weight rods with 48-inch outer casing, etc.). Currently, standard Geoprobe® 1.25-inch probe rods must be used with 24-inch, 36-inch, and 1-meter MC5 Sample Tubes.

A weight reduction of up to 64% is provided by the 1.25-inch Light-Weight Center Rods over standard 1.25-inch probe rods. As a result, considerably less energy is expended when retrieving the 1.25-inch Light-Weight Center Rods from within the outer casing during operation of the MC5 System.

Sample Liners
Sample liners are made of heavy-duty clear plastic for convenient inspection of the soil sample. Nominal lengths of 24 inches, 36 inches, 1 meter, 48 inches, and 60 inches are available. Choose the liner length corresponding to the length of the sample tube used (e.g. 60-inch liners with 60-inch sample tubes).

Cutting Shoes
Six cutting shoes are available for use with the MC5 Soil Sampling System (Fig. 3.2). The extended shank cutting shoes (23978, 28237, and 26078) fit inside the sample liner and help soil pass freely into the liner. The other three cutting shoes (22922, 23957, 29552) require an MC Core Catcher (AT8531) or MC Spacer Ring (AT8532) in order to properly connect to the sample liners.

The most prominently used cutting shoes are the two "standard" cutting shoes (22922 and 23978). These cutting shoes collect a 1.5-inch (38-mm) diameter soil core.

Undersized cuttings shoes (23957, 28237, and 29552) collect a smaller 1.35-inch (34-mm) soil core and are used in formations with plastic clays or other soil types that lead to overfilling of the sampler liner. Of these, the 29552 and 28237 cutting shoes are also thicker at the leading end for increased durability in harsh conditions where cobbles or large gravel are present.

Soil formations with highly plastic clays may call for an even smaller soil core. In these conditions, a 26078 cutting shoe with its 1.25-inch (32-mm) soil core is most effective.

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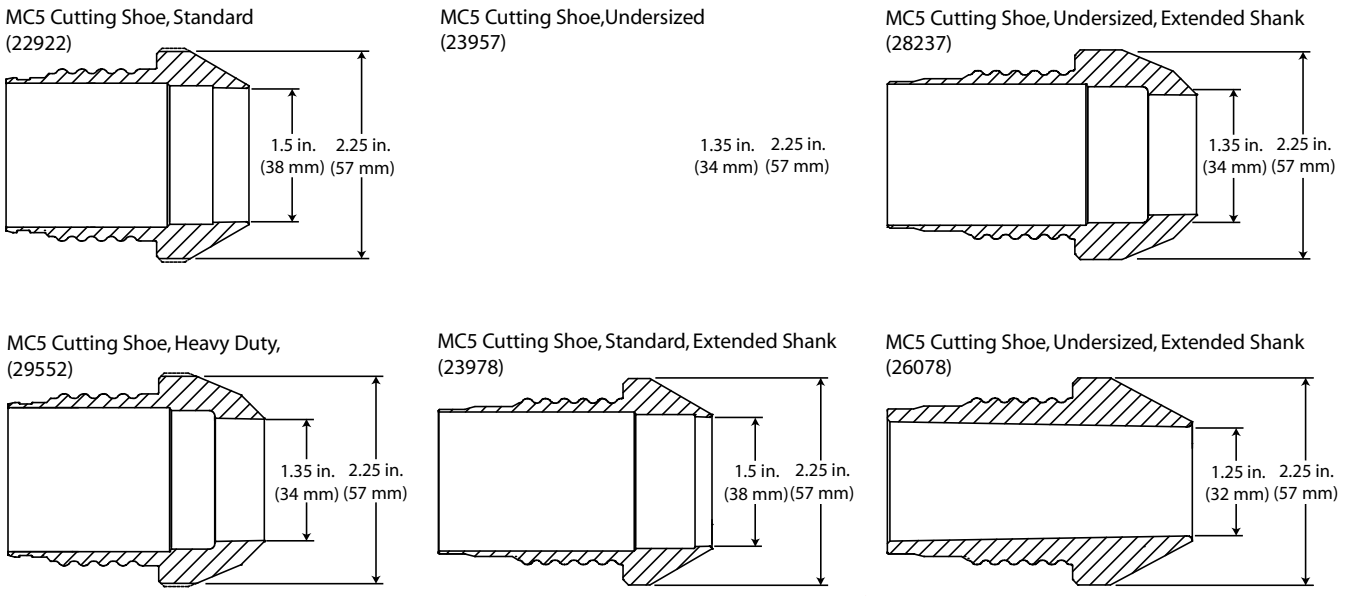

Figure 3.2
Cutting Shoe Options for Macro-Core® MC5 Soil Sampler

4.0 OPERATION

All parts shown in illustrations are those most commonly used configuration for the MC5 Sampling System. Refer to Section 3.0 for part numbers and additional tooling options.

4.1 Decontamination

Before and after each use, thoroughly clean all parts of the soil sampling system according to project requirements. Parts should be inspected for wear or damage at this time. During sampling, a clean new liner is used for each soil core.

Cleaning inside the probe rods and MC5 sample tubes is accomplished with the nylon brushes and extension rods listed in Appendix A. Thread a nylon brush and handle onto an extension rod of suitable length. Using clean water and phosphate-free soap, cycle the brush inside the probe rod or sample tube to remove contaminants. Rinse with clean water and allow to air dry.

4.2 Field Blank

It is suggested that a field blank be taken on a representative sample liner prior to starting a project and at regular intervals during extended projects. Liners can become contaminated in storage. A field blank will prove that the liners do not carry contaminates which can be transferred to soil samples. The following information is offered as an example method which may be used to take a field blank. Make the appropriate modifications for the specific analytes of interest to the investigation.

Example Procedure Required Equipment

MC Liner ..... (1)
MC Vinyl End Caps ..... (2)
Distilled Water ..... (100 ml)
VOA Vial (or other appropriate sample container) ..... (1)

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1. Place a vinyl end cap on one end of the liner.
2. Pour 100 milliliters of distilled water (or other suitable extracting fluid) into the liner.
3. Place a vinyl end cap on the open end of the liner.
4. From the vertical position, repeatedly invert the liner so that the distilled water contacts the entire inner surface. Repeat this step for one minute.
5. Remove one end cap from the liner, empty contents into an appropriate sample container, and cap the container.
6. Perform analysis on the extract water for the analytes of interest to the investigation.

4.3 Open-Tube Sampler Assembly

Figure 4.1. The spacer ring fits securely onto the MC5 Cutting Shoe.

1a. Using the MC Core Catcher

Place the open end of an MC Core Catcher over the threaded end of an MC5 Cutting Shoe (22992, 23957, 29552) as shown in Figure 4.2. Apply pressure to the core catcher until it snaps into the machined groove on the cutting shoe. The core catcher should be used in loose soils, especially saturated sands (non-cohesive soils). Use of the core catcher is not necessary in tough, cohesive soils or tight clays, and may interfere with sampling especially in soft clays. The "fingers" of the core catcher flex outward to let soil move into the liner while sampling.

1b. Using the MC Spacer Ring

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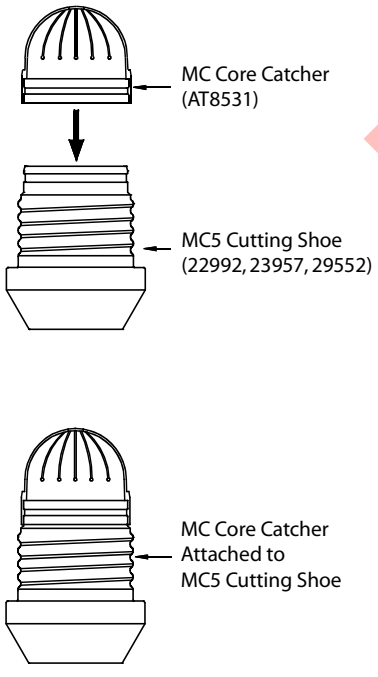

Figure 4.2
Core Catcher Attachment

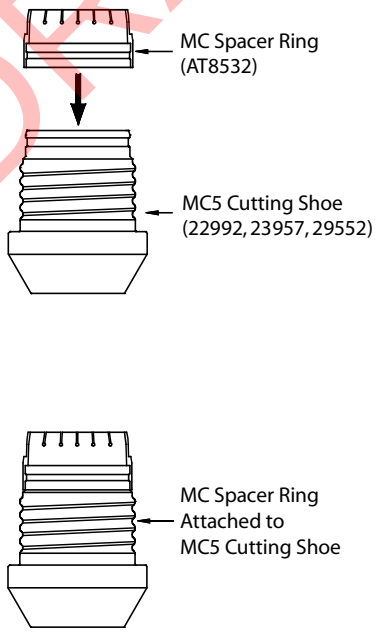

Figure 4.3
Spacer Ring Attachment

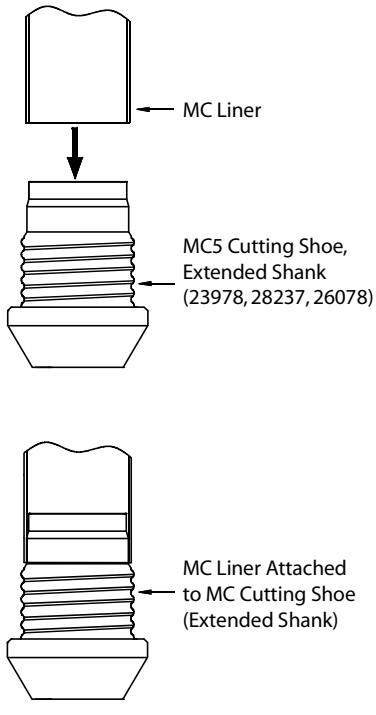

Figure 4.4
Liner Attachment on Extended Shank

Push the base of an MC Spacer Ring onto the threaded end of an MC5 Cutting Shoe (22992, 23957, 29552) until it snaps into the machined groove on the cutting shoe (Fig. 4.1 and Fig. 4.3). Spacer rings should be used when sampling cohesive soils. It allows soil to pass freely over the junction between the liner and cutting shoe.

1c. Using the Extended Shank Cutting Shoe

The cutting shoes with extended shanks (23978, 28237, 26078) do not use core catchers or spacer rings. MC5 Liners should securely slide onto the end of these cutting shoes (Fig. 4.4). The extended shank cutting shoes should only be used when sampling cohesive soils. When sampling loose soils, especially saturated sands (non-cohesive soils), a cutting shoe with an MC Core Catcher is recommended.

2. Place either end of the liner onto the spacer ring or core catcher (Fig. 4.6). If you are using a cutting shoe with an extended shank, do not use a spacer ring or core catcher (Fig. 4.7). The liner should fit securely onto the spacer ring, core catcher, or cutting shoe.

3. Slide whole assembly into either end of the sample tube (Fig. 4.8). Thread the cutting shoe onto the sample tube (Fig. 4.9). If the thread is clean, it should easily thread on by hand. In some cases, a wrench may be necessary for tightening. There shouldn't be a gap between the cutting shoe and sample tube.

4. Thread an MC5 Drive Head into the top of the sample tube (Fig. 4.10). Securely tighten the drive head by hand. Ensure that the end of the sample tube contacts the machined shoulder of the drive head.

Sampler Assembly is Complete

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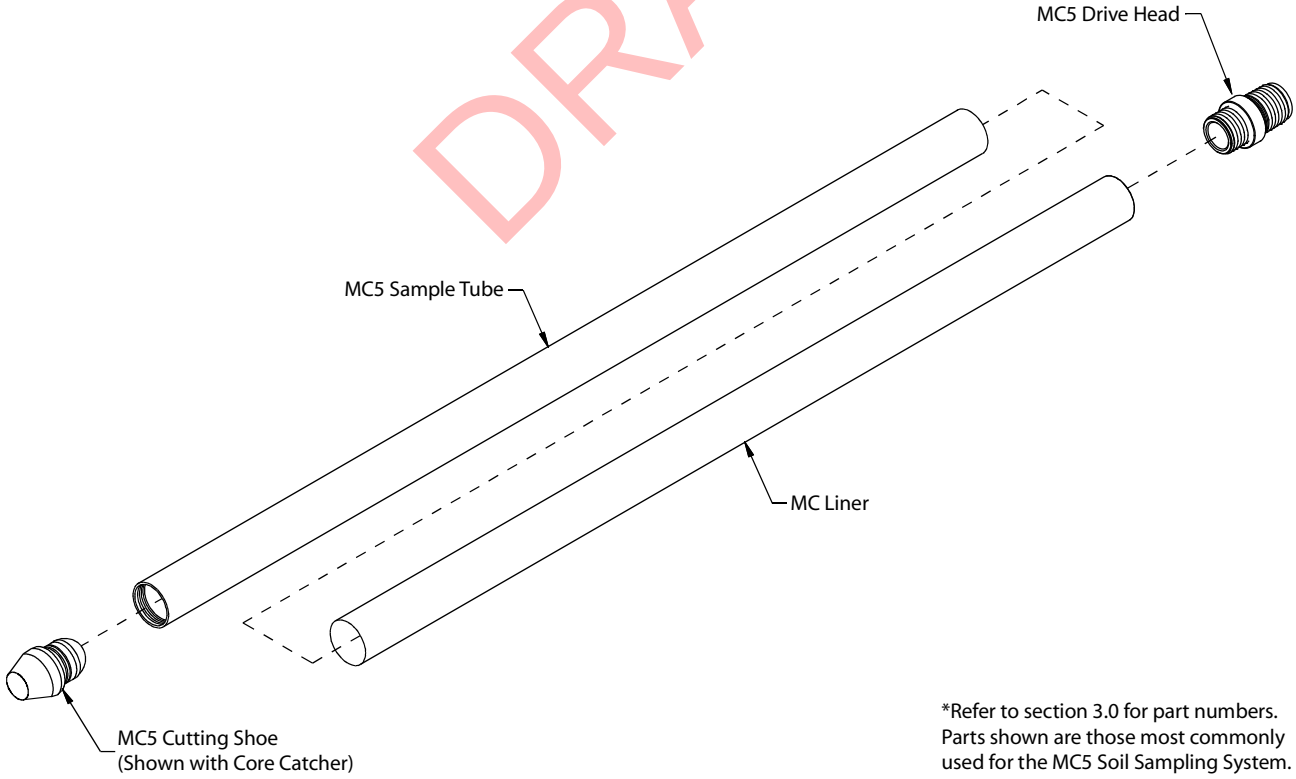

Figure 4.5
MC5 Open-Tube Sampler Assembly

Figure 4.6. Place either end of the liner onto the spacer ring or core catcher. The liner should fit securely.

Figure 4.7. Place either end of the liner onto the extended shank cutting shoe. (This is used in place of a spacer ring or core catcher)

Figure 4.8. Slide whole assembly into either end of the sample tube.

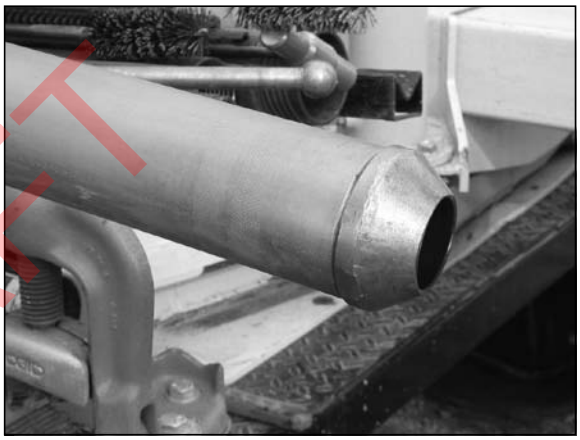

Figure 4.9. Thread the cutting shoe onto the sample tube.

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Figure 4.10. Thread the MC5 Drive Head onto the opposite end of the sample tube. Tighten by hand.

4.4 MC5 Closed-Point Sampler Assembly

The Macro-Core® 1.25-inch Light-Weight Center Rod Sampling System seals the leading end of the sampler with a point (Fig. 4.11) assembly that is held in place with a 1.25-inch light weight center rod. Once advanced to the top of the sampling interval, the 1.25-inch Light-Weight Center Rods are removed from the probe rod string.

1. Install an O-ring in the machined groove on the piston rod point (Fig. 4.12).

2. Push the MC5 Closed Piston Point (28113 or 26865) completely into the cutting shoe as shown in Figure 4.12. Note that the standard point (28113) is used with 1.5-inch (38-mm) ID cutting shoes and the undersized point (26865) is for cutting shoes with a 1.35-inch (34-mm) ID.

Figure 4.11. The MC5 Closed Piston Point slides into the cutting shoe.

3a. Using the MC Core Catcher

Place the open end of an MC Core Catcher over the threaded end of an MC5 Cutting Shoe (22992, 23957, 29552) as shown in Figure 4.13. Apply pressure to the core catcher until it snaps into the machined groove on the cutting shoe. The core catcher should be used in loose soils, especially saturated sands (non-cohesive soils). Use of the core catcher is not necessary in tough, cohesive soils or tight clays, and may interfere with sampling especially in soft clays. The "fingers" of the core catcher flex outward to let soil move into the liner while sampling.

3b. Using the MC Spacer Ring

Push the base of an MC Spacer Ring onto the threaded end of an MC5 Cutting Shoe (22992, 23957, 29552) until it snaps into the machined groove on the cutting shoe (Fig. 4.14). Spacer rings should be used when sampling cohesive soils. It allows soil to pass freely over the junction between the liner and cutting shoe.

3c. Using the Extended Shank Cutting Shoe

The cutting shoes with extended shanks (23978, 28237) do not use core catchers or spacer rings. MC5 Liners should securely slide onto the end of these cutting shoes (Fig. 4.15). The extended shank cutting shoes shoud only be used when sampling cohesive soils. When sampling loose soils, especially saturated sands (non-cohesive soils), a cutting shoe with an MC Core Catcher is recommended.

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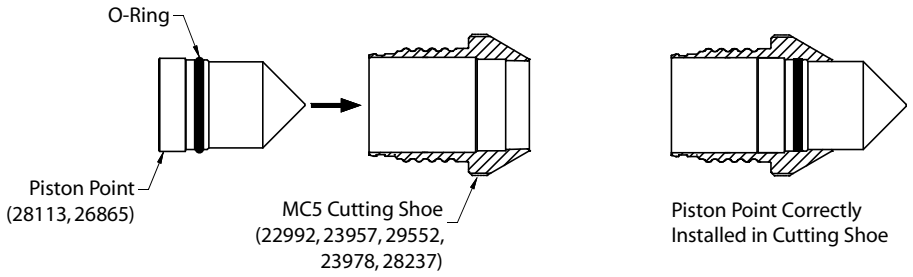

Figure 4.12
Installation of MC5 Closed Piston Point Assembly in MC5 Cutting Shoe

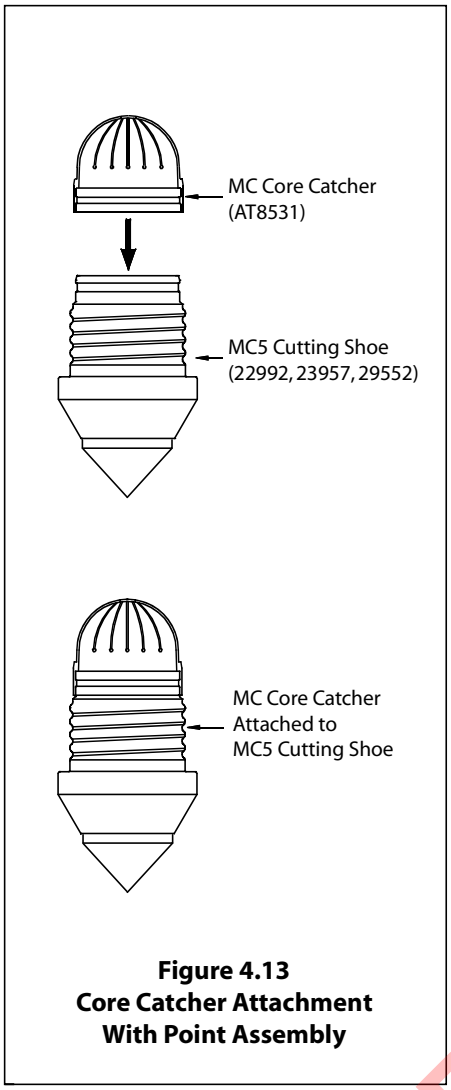

Figure 4.13
Core Catcher Attachment
With Point Assembly

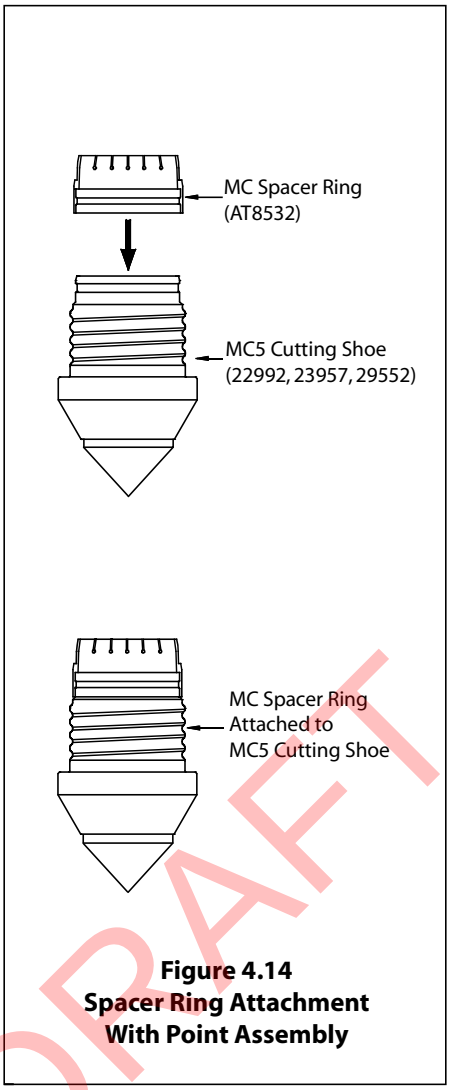

Figure 4.14
Spacer Ring Attachment
With Point Assembly

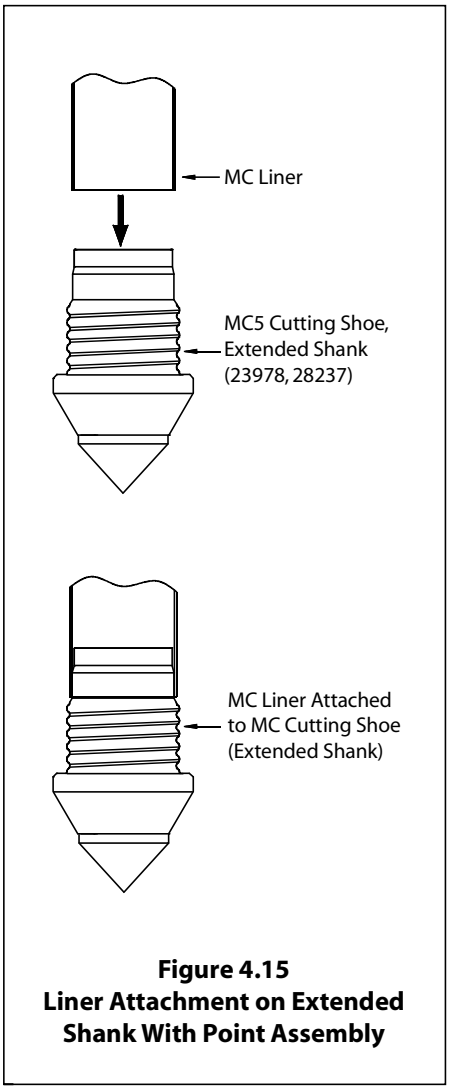

Figure 4.15
Liner Attachment on Extended
Shank With Point Assembly

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Refer to Figure 4.16 for MC5 Closed-Point Sampler Assembly

4. Place either end of the liner onto the spacer ring or core catcher (Fig. 4.18). If you are using a cutting shoe with an extended shank, do not use a spacer ring or core catcher (Fig. 4.19). The liner should fit securely onto the spacer ring, core catcher, or cutting shoe.

5. Slide whole assembly into either end of the sample tube (Fig. 4.20). Thread the cutting shoe onto the sample tube (Fig. 4.21). If the thread is clean, it should easily thread on by hand. In some cases, a wrench may be necessary for tightening. There shouldn't be a gap between the cutting shoe and sample tube.

6. Thread an MC5 Drive Head into the top of the sample tube. Securely tighten the drive head by hand. Ensure that the end of the sample tube contacts the machined shoulder of the drive head (Refer to Figure 4.10).

continued on page 14

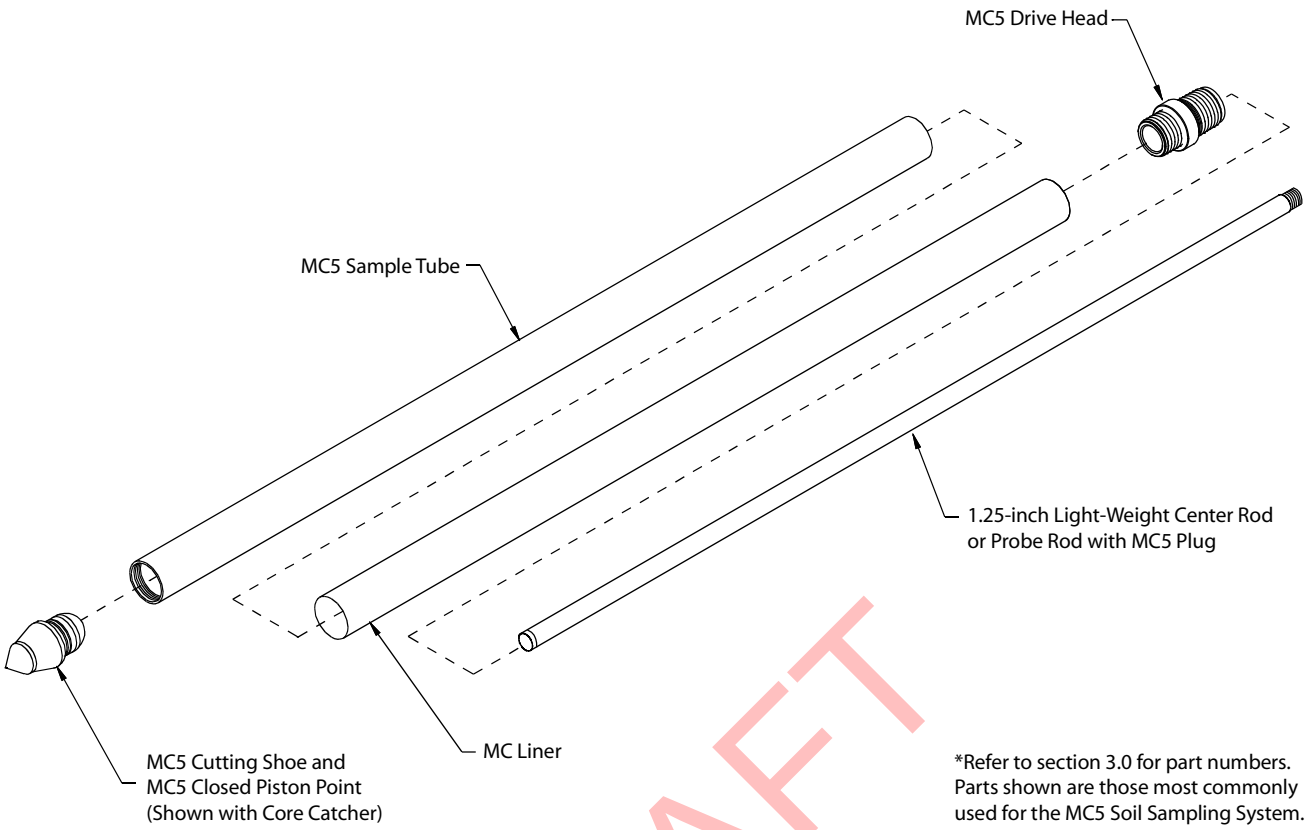

Figure 4.16
MC5 Closed-Point Sampler Assembly

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7. Thread an MC5 Plug (23641) onto 1.25-inch light-weight center rod (Fig. 4.22). Note that light-weight center rods are only available in 48-inch and 60-inch lengths. Utilize 1.25-inch probe rods if other lengths are required.

8. Insert the light-weight center rod and MC5 Plug into sample tube assembly (Fig. 4.23), sending the plug end in first. Allow it to come in contact with the top of the Piston Point (Fig. 4.17).

Sampler Assembly is Complete

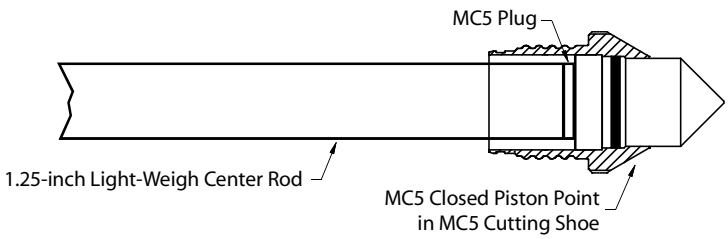

Figure 4.17
MC5 Closed-Point Center Rod Assembly

Figure 4.18. Place either end of the liner onto the spacer ring or core catcher. The liner should fit securely.

Figure 4.19. Place either end of the liner onto the extended shank cutting shoe. (This is used in place of a spacer ring or core catcher)

Figure 4.20. Slide whole assembly into either end of the sample tube.

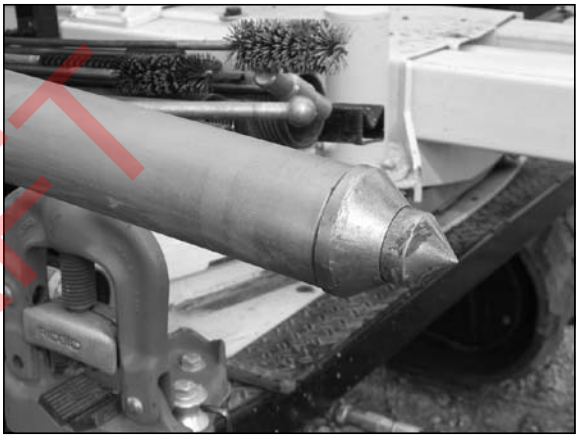

Figure 4.21. Thread the cutting shoe and point onto the sample tube.

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Figure 4.22. The MC5 Plug is threaded onto the end of the 1.25-inch light-weight center rod.

Figure 4.23. The MC5 Plug and a 1.25-inch light-weight center rod are inserted into the sample tube.

4.7 Open-Tube Sampling

The MC5 Open-Tube Sampler is used to gather continuous soil cores beginning from ground surface. A representative soil sample is obtained by driving the assembled sampler one sampling interval into the subsurface through undisturbed soil. Upon retrieving the sampler, the liner and soil core are removed. The sampler is then properly decontaminated, reassembled with a new liner, and inserted back down the same hole to collect the next soil core.

Instructions for operating the MC5 Open-Tube Sampler are given in this section.

1. Place a drive cap onto the drive head (Fig. 4.24) of an assembled Open-Tube Sampler (Refer to Section 4.3 for sampler assembly).

2. Raise the probe unit hammer assembly to its highest position by fully extending the probe cylinder.

3. Position the MC5 Sampler directly under the hammer with the cutting shoe centered between the toes of the probe foot. The sampler should now be parallel to the probe derrick. Step back from the unit and visually check sampler alignment (Fig. 4.25).

4. Apply static weight and hammer percussion to advance the sampler until the drive head reaches the ground surface. (Fig. 4.27A)

   NOTE: Activate hammer percussion whenever collecting soil. Percussion helps shear the soil at the leading end of the sampler so that it moves into the sample tube for increased recovery.

5. Raise the hammer assembly a few inches to provide access to the top of the sampler.

6. Remove the drive cap and thread a pull cap onto the sampler drive head (Fig. 4.26).

7. Lower the hammer assembly and hook the hammer latch over the pull cap. Raise the hammer assembly to pull the sampler completely out of the ground. If a winch is available, it can be used with a pull plate to retract the tool string. A Rod Grip Pull Handle can also be used to retract the tool string.

8. Proceed to Section 4.9 for instructions on recovering the soil core from the MC5 Sampler.

To sample consecutive soil cores, advance a clean sampler down the previously opened hole (Fig. 4.27B) to the top of the next sampling interval (Fig. 4.27C). Drive the tool string the length of the sampler to collect the next soil core (Fig. 4.27D). Switch to an MC5 Center Rod Sampler if excessive side slough is encountered.

NOTE: Use caution when advancing or retrieving the sampler within an open hole. Low side friction may allow the sampler and probe rods to drop down the hole when released. To prevent equipment loss, hold onto the tool string with a pipe wrench when needed.

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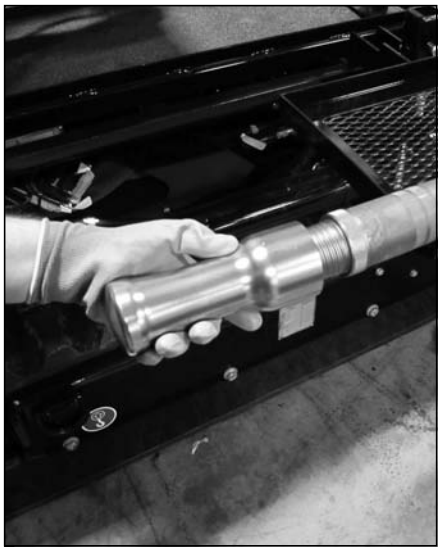

Figure 4.24. Place drive cap onto sampler drive head.

Figure 4.25. The sampler should be parallel to the probe derrick for driving.

Figure 4.26. The pull cap is one way to remove the sampler from the ground.

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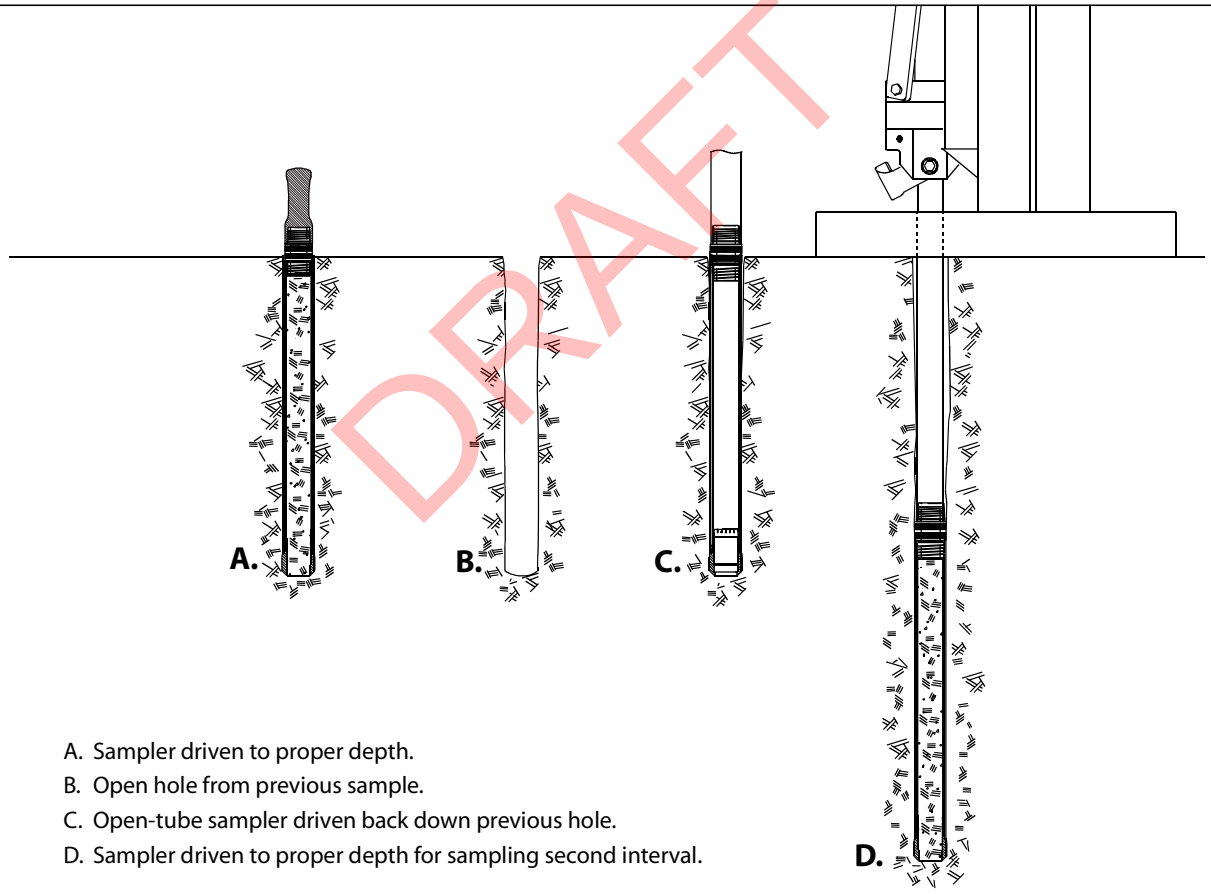

A. Sampler driven to proper depth.
B. Open hole from previous sample.
C. Open-tube sampler driven back down previous hole.
D. Sampler driven to proper depth for sampling second interval.

Figure 4.27
Phases of MC5 Open-Tube Soil Sampling

4.8 Closed-Point Sampling with the MC5 Center Rod System

Material collapsing from the probe hole sidewall can make it difficult to collect representative soil cores from significant depths with an open-tube sampler. To overcome this problem, the MC5 Sampler can be equipped with a center rod assembly that will hold the piston point in place. This allows the sealed sampler to pass through the slough material and then it can be opened at the appropriate sampling interval.

Instructions for operating the MC5 Closed-Point Sampler are given in this section.

1. Place a drive cap onto the center rod and a drive cap onto the drive head of an assembled Closed-Point Sampler (Refer to Section 4.4 for sampler assembly).

2. Raise the probe unit hammer assembly to its highest position by fully extending the probe cylinder.

3. Position the MC5 Sampler directly under the hammer with the cutting shoe centered between the toes of the probe foot. The sampler should now be parallel to the probe derrick. Step back from the unit and visually check sampler alignment (Fig. 4.25).

4. Apply static weight and hammer percussion to advance the sampler until the drive head reaches the ground surface (Fig. 4.28A).

5. Add additional probe rods and 1.25-inch light-weight center rods to the tool string until the desired sampling interval is reached (Fig. 4.28B).

6. Once the sampling interval is reached, remove the center rod string (Fig. 4.28C).

7. Add an additional probe rod to the string and place a drive cap on the probe rod (Fig. 4.28D).

8. Advance the tool string to collect the soil core in the liner (Fig. 4.28E).

   NOTE: Activate hammer percussion whenever collecting soil. Percussion helps shear the soil at the leading end of the sampler so that it moves into the sample tube for increased recovery.

9. Lower the hammer assembly and hook the hammer latch over the pull cap. Raise the hammer assembly to pull the first probe rod out of the ground. Remove the rod and place the pull cap on the next rod of the tool string. Continue pulling probe rods until the MC5 Sampler is brought to the ground surface. If a winch is available, it can be used with a pull plate to retract the tool string. An RG Handle is another option to retract the tool string.

   NOTE: Use caution when advancing or retrieving the sampler within an open hole. Low side friction may allow the sampler and probe rods to drop down the hole when released. To prevent equipment loss, hold onto the tool string with a pipe wrench when needed.

10. Proceed to Section 4.9 for instructions on recovering the soil core from the MC5 Sampler.

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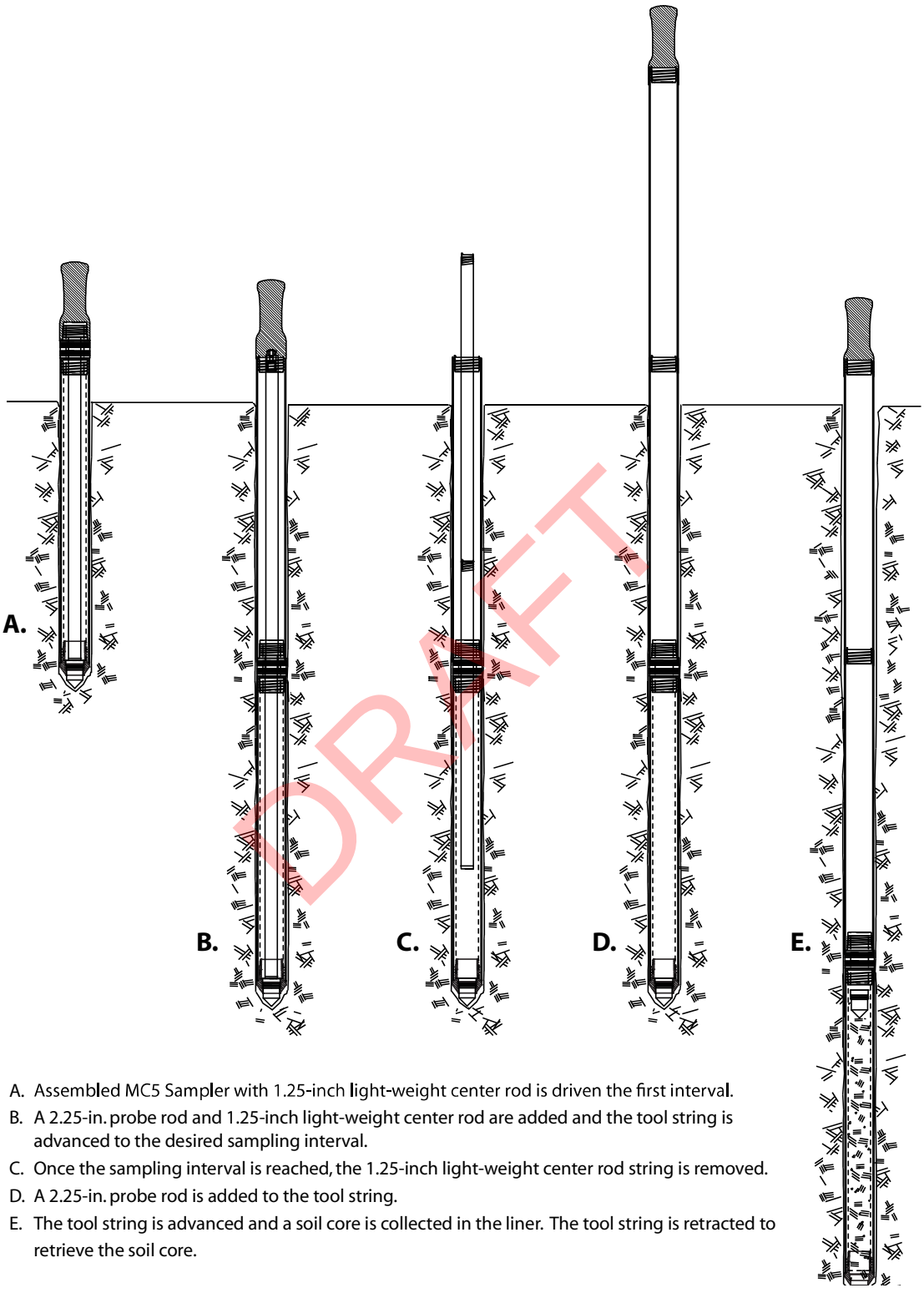

A. Assembled MC5 Sampler with 1.25-inch light-weight center rod is driven the first interval.
B. A 2.25-in. probe rod and 1.25-inch light-weight center rod are added and the tool string is advanced to the desired sampling interval.
C. Once the sampling interval is reached, the 1.25-inch light-weight center rod string is removed.
D. A 2.25-in. probe rod is added to the tool string.
E. The tool string is advanced and a soil core is collected in the liner. The tool string is retracted to retrieve the soil core.

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Figure 4.28
Phases of MC5 Closed-Point Center Rod System

4.9 Soil Core Recovery

The soil sample is easily removed from the MC5 Sampler by unthreading the cutting shoe and pulling out the liner (Fig. 4.29). A few sharp taps on the cutting shoe with a pipe wrench will often loosen the threads sufficiently to allow removal by hand. If needed, the exterior of the cutting shoe features wrench flats for attaching a wrench to loosen tight threads. With the cutting shoe removed, simply pull the liner and soil core from the sample tube (Fig. 4.31). A Hydraulic Liner Extruder is also available for mounting on your machine to remove liners (Fig. 4.30).

If the closed-point sampler is used, the piston point is now retrieved from the end of the liner (Fig. 4.32). Secure the soil sampler by placing a vinyl end cap on each end of the liner.

Undisturbed soil samples can be obtained from liners by splitting the liner. The MC Liner (AT8010) is used to make longitudinal cuts along the liner (Fig. 4.33).

Figure 4.29. Remove the MC5 Cutting Shoe and liner from the MC5 Sampler Tube.

Figure 4.30. The Hydraulic Liner Extruder helps remove the liner.

Figure 4.31. MC5 Liner filled with soil core.

Figure 4.32. MC5 Closed Piston Point is retrieved from the top of the liner.

Figure 4.33. MC Liner Cutter makes two longitudinal cuts in PVC Liners.

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4.10 Tips to Maximize Sampling Productivity

The following suggestions are based on the collective experiences of Geoprobe® operators:

1. Organize your truck or van. Assign storage areas to all tools and equipment for easy location. Transport sample tubes, probe rods, 1.25-inch light-weight center rods, and liners in racks. Above all, minimize the number of items lying loose in the back of your vehicle.

2. Take three or four samplers to the field. This allows the collection of several samples before stopping to clean and decontaminate the equipment. A system is sometimes used where one individual operates the probe while another marks the soil cores and decontaminates the used samplers.

3. A machine vise is recommended. With the sampler held in a vise, the operator has both hands free to remove the cutting shoe, drive head, and sample liner. Cleanup is also easier with both hands free. Geoprobe® offers an optional machine vise (FA300).

4. Organize your worksite. Practice with the sampler to identify a comfortable setup and then use the layout whenever sampling. A collapsible table or stand is handy to hold decontaminated sampler tubes and liners. Equipment may also be protected from contamination by placing it on a sheet of plastic on the ground.

   Instead of counting probe rods for each trip in-and-out of the probe hole, identify separate locations for "new" rods and "used" rods. Collect the first sample from the open hole using "new" rods. As each probe rod is removed during sampler retrieval, place it in the "used" rod location. Now advance a clean sampler back down the same hole using all of the rods from the "used" location. Add one "new" rod to the string and then drive the tools to collect the next soil core. Once again, remove each probe rod and place it in the "used" rod location as the sampler is retrieved. Repeat this cycle using all the "used" rods to reach the bottom of the probe hole, and one "new" rod to fill the sampler.

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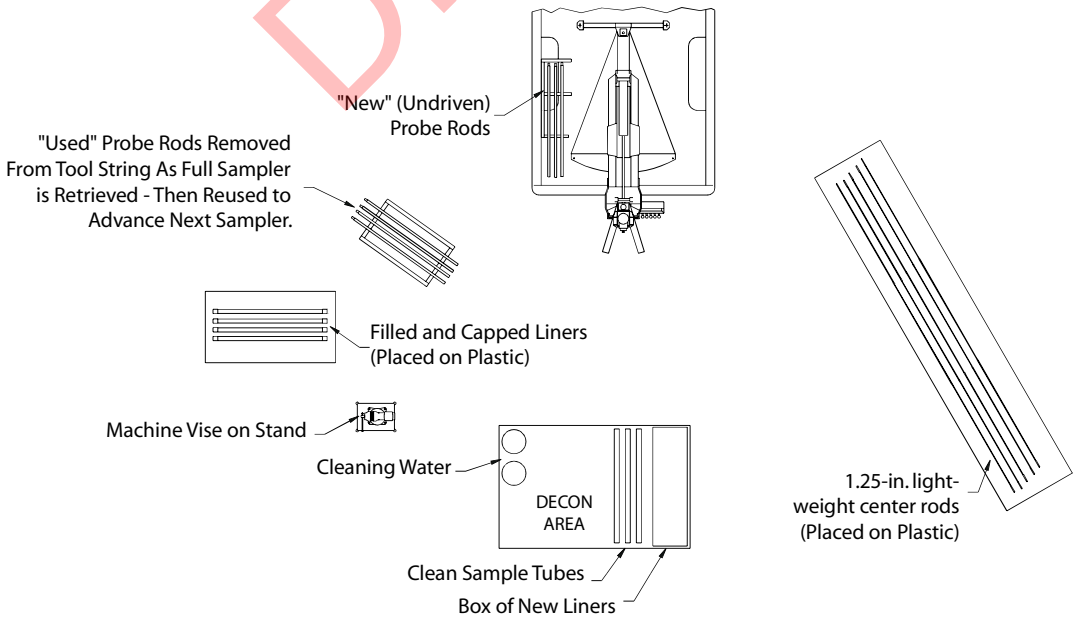

Figure 4.36
Equipment Layout Example to Maximize Sampling Productivity

5. Cleanup is very important from the standpoint of operation as well as decontamination. Remove all dirt and grit from the threads of the drive head, cutting shoe, and sample tube with a nylon brush (BU700). Without sufficient cleaning, the cutting shoe and drive head will not thread completely onto the sample tube and probe rods. The threads may be damaged if the sampler is driven in this condition.

   Ensure that all soil is removed from inside the sample tube. Sand particles are especially troublesome as they can bind liners in the sampler. Full liners are difficult to remove under such conditions. In extreme cases, the soil sample must be removed from the liner before it can be freed from the sample tube.

5.0 REFERENCES

Geoprobe Systems®, 2003. *Tools Catalog, V. 6.*

APPENDIX A
ALTERNATIVE PARTS

Geoprobe® Tools and Equipment	Part Number
Drive Cap, GH40 Series, Threaded, 2.25 in	25362
Drive Cap, GH60 Series, Threaded, 2.25 in	25363
Drive Cap, GH60 Series, Threaded, 2.125 in.	15673
Nylon Brush, Macro-Core® Tool	BU700
Nylon Brush, 2.25-in. and 2.125-in. probe rods	BU2125
Extension Rod Handle	AT69
Extension Rod (60-in.)	10073
Extension Rod (48-in.)	AT671
Extension Rod (36-in.)	AT67

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Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.

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Geoprobe Systems®

A DIVISION OF KEJR, INC.

Corporate Headquarters
1835 Wall Street • Salina, Kansas 67401
1-800-GEOPROBE (1-800-436-7762) • Fax (785) 825-2097
www.geoprobe.com

cox environmental

CES-SOP-02: Monitoring Well Installation, Development, Purging, and Sampling

All monitoring wells will be developed per the following Alaska Department of Environmental Conservation (ADEC) guidance documents:

- **Monitoring Well Guidance (ADEC, 2017)**
- **Field Sampling Guidance (ADEC, 2017)**
- **Underground Storage Tanks Procedures Manual: Guidance for Treatment of Petroleum-Contaminated Soil and Groundwater and Standard Sampling Procedures (ADEC, 2017)**

Monitoring Well Installation

Groundwater monitoring serves several purposes, such as ambient monitoring, source monitoring, case preparation monitoring, and research monitoring (Barcelona et al., 1985). Wells installed for each of these purposes must satisfy different requirements, and may require different strategies for well design and installation. Prior to design and installation there should be a clear understanding of what the monitoring program is intended to accomplish. Is the monitoring well intended for site characterization or plume delineation, long term monitoring, contaminant screening, final compliance with cleanup standards for site closure, product recovery or a remedial action, or some other purpose?

Selection of monitoring well type, materials, and installation method is a site-specific determination. Site logistics and economics often influence choices. Locations without road access can be logistically challenging and incur increased project costs for site investigation. In all cases, clearly identify project objectives in a work plan developed in consultation with ADEC.

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Proper well spatial and vertical location is critical to ensure accurate monitoring of the groundwater flow regime. Monitoring wells and well points are typically installed in the uppermost permeable water-bearing zone under or adjacent to a regulated facility or potential source of contamination. Consider natural, seasonal, and anthropogenic fluctuations in water table elevation in determining the well location. Natural fluctuations are typically due to infiltration of snowmelt or precipitation, proximity to rivers with seasonal high water levels, or tidal fluctuations. Anthropogenic fluctuations can result from pumping, wastewater disposal, or paving to decrease infiltration rates. Consider the behavior of a contaminant plume over distance to ensure that placement and construction of monitoring wells is appropriate (Wiedemeier et al., 1999).

Well design and installation must be appropriate to ensure that groundwater samples and water level measurements characterize discrete stratigraphic intervals. Location of the screened interval relative to the water table elevation may influence sampling results. For example, a well screened at the water table, with some screen above the water table and some below the water table, will intercept floating petroleum product; a well with the top of the screened interval located below the water table will not intercept floating petroleum product under static conditions. Well design and installation must prevent the introduction of surface contaminants into the groundwater and prevent leakage of groundwater or contaminants between stratigraphic intervals in the well bore or along the well annulus. If the well leaks, correct the leak or decommission the well. Do not install monitoring wells in locations where they are subject to periodic or seasonal inundation by floodwaters, unless the well has special watertight construction. Protect monitoring wells from loss of integrity by soil erosion, soil settlement, shrink-swell soil conditions, frost heaving of soils, damage by vehicles or heavy equipment, and other site-specific hazards. Completion of monitoring wells at- or below -grade is less preferable than above-grade completions due to the potential for surface water infiltrating the monitoring well casing.

A drilled, long-term monitoring well is generally composed of well casing, well screen, and filter pack (Figure 1). Construct monitoring wells with new materials that will not physically, chemically, or biologically affect the groundwater quality, or be deleteriously affected by the subsurface environment. The well screen is an intake where groundwater can flow into the well; the filter pack surrounds the well screen. Install the well in an open borehole created by advancing a soil boring, usually with a hollow-stem auger drill rig. Advance the soil boring until the soil core (s) demonstrates saturated soil conditions, indicating that the groundwater table has been encountered. After the water table has been identified in the soil boring, remove the drill rods from the open borehole and install the monitoring well.

Survey monitoring wells vertically and horizontally. Survey the top of the well casing and ground surface for use as a reference point to determine water-level elevations and sampling depths and to evaluate groundwater flow direction. All survey data must be recorded in the field notes and submitted with the report. The location survey must achieve a horizontal accuracy of 1.0 feet, and the elevation surveys must achieve a vertical accuracy of 0.01 foot. Sites undergoing contaminant assessment monitoring must have the wells surveyed as described above, and re-survey monitoring wells every year, unless otherwise approved by ADEC on a site-specific basis. Based on site conditions ADEC may require that a survey be completed by a registered professional surveyor or registered professional engineer.

For accurate water level measurements, permanently mark the monitoring well with a reference point on the actual monitoring well casing, not the outer surface casing. Permanently attach a facility or project-unique identification number on the inner and outer well casings. All well construction logs with soil boring information are required to be submitted to the Alaska Department of Natural Resources Division of Mining Land and Water in accordance with 11 AAC 93.140(g).

Also, submit documentation of the well design, well construction logs, and the materials used to ADEC. This information is useful for determining if the monitoring well design, installation, or history may be affecting sampling results or the interpretation of site conditions.

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General Construction Procedures Key Principals, Specifications, and Precautions

1. Properly decontaminate well construction materials prior to installation.
2. Prevent contamination when joining casings and attaching the screen.
3. For long-term monitoring wells, place the filter pack into the annulus to a minimum of two feet above the top of the screen and one foot beneath the well end cap.
4. Use bottom caps or end plugs.
5. Use permanent or temporary surface casing if contamination or sloughing is a potential issue (drill augers should never be removed from the hole without concurrently filling borehole voids with appropriate sealant media).
6. For long-term monitoring wells, reduce the required filter pack height to allow for annular space sealant.
7. For long-term monitoring wells following installation, “sound” the filter pack for proper placement.
8. For long-term monitoring wells apply grout or bentonite chips to seal the annular space.
9. If the borehole or monitoring well is advanced through an aquitard, the penetration through the aquitard must be sealed at the same interval using grout or bentonite chips, unless otherwise approved by ADEC.
10. For all wells, pour grouts or slurries freely with or without the use of a tremie pipe.

11. Take appropriate precautions during drilling to avoid introducing contaminants into the well. Prevent vertical movement of water or contaminants between waterbearing zones in either the boring or the well annulus.
12. Avoid using drilling mud, synthetic drilling fluids, or petroleum- or metal-based pipe joint compounds and other potential contaminants unless necessary.
13. If it is necessary to add water during drilling, use only potable water and first identify the water source.
14. If it is necessary to add drilling mud to stabilize the hole or control down-hole fluid losses, use only high yield sodium bentonite clay free of all organic polymer additives.
15. Properly decontaminate all equipment placed into the well by steam cleaning, high-pressure hot water, or similar methods between well installations.
16. Manage cuttings, or water, removed from the well in accordance with 18 AAC 75 or 18 AAC 78.
17. Complete an "as built" drawing/schematic for each constructed monitoring well.
18. Survey wells vertically and horizontally with survey loops that close within 0.01 foot vertically, and 1.0 feet horizontally. The well survey data must be provided to ADEC in a written report. Submit a record of the well design, installation, and the materials used to ADEC.
19. Install a cement or asphalt surface seal, where appropriate.

Procedures for Specific Types of Wells

Drilled Wells - Key Principals, Specifications, and Precautions

1. Select the proper drill rig.
2. Evaluate site-specific hydrogeologic information from all available sources, including the physical and chemical properties of the groundwater and any contaminants known or suspected to be present in the groundwater.
3. Develop a conceptual hydrogeologic model of the site.
4. Determine screened interval.
5. Determine the diameter of the well.
6. Determine the proper inside diameter of the borehole (at least 4 inches larger than the riser and screen diameter).
7. Take appropriate precautions during drilling to avoid introducing contaminants into the borehole.
8. Proceed with soil recovery per the ADEC approved Work Plan.
9. Complete an "as built" drawing/schematic for each constructed monitoring well.
10. Avoid using drilling mud, synthetic drilling fluids, or petroleum- or metalbased pipe joint compounds and other potential contaminants unless necessary.
11. If it is necessary to add water to the borehole during drilling, use only potable water and first identify the water source.
12. If it is necessary to add drilling mud to the borehole during drilling to stabilize the hole or control down-hole fluid losses, use only high yield sodium bentonite clay free of all organic polymer additives.
13. Properly decontaminate all equipment placed into the borehole by steam cleaning, high-pressure hot water, or similar methods before and after use at the site and between boreholes.
14. Manage cuttings, or water, removed from the borehole in accordance with 18 AAC 75 or 18 AAC 78.
15. Survey wells vertically and horizontally with survey loops that close within 0.01 foot vertically, and1.0 foot horizontally. The well survey data must be provided to ADEC in a written report. Submit a record of the well design, installation, and the materials used to ADEC.

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Direct Push Wells - Key Principals, Specifications, and Precautions

1. Determine the purpose of the well.
2. Evaluate site-specific hydrogeologic information from all available sources, including the physical and chemical properties of the groundwater and any contaminants known or suspected to be present in the groundwater.
3. Develop a conceptual hydrogeologic model of the site.
4. Determine screened interval.
5. Determine the diameter of the well.
6. Take appropriate precautions during installation to avoid introducing contaminants into the well. Prevent vertical movement of water or contaminants between water-bearing zones in either the boring or the well annulus.
7. Properly decontaminate all equipment placed into the well by steam cleaning, high-pressure hot water, or similar methods between well installations.
8. Manage cuttings, or water, removed from the well in accordance with 18 AAC 75 or 18 AAC 78.
9. Complete an "as built" drawing/schematic for each constructed monitoring well.
10. Survey wells vertically and horizontally with survey loops that close within 0.01 foot vertically, and 1.0 foot horizontally. The well survey data must be provided to ADEC in a written report. Submit a record of the well design, installation, and the materials used to ADEC.

Excavation Installed Wells - Key Principals, Specifications, and Precautions

1. Determine the purpose of the well.
2. Evaluate site-specific hydrogeologic information from all available sources, including the physical and chemical properties of the groundwater and any contaminants known or suspected to be present in the groundwater.
3. Develop a conceptual hydrogeologic model of the site.
4. Determine screened interval based on known groundwater levels.
5. Determine the diameter of the well.
6. Take appropriate precautions during excavation/placement to avoid introducing contaminants into the well. Prevent vertical movement of water or contaminants between water-bearing zones.
7. Properly decontaminate all equipment placed into the well by steam cleaning, high-pressure hot water, or similar methods between well installations.
8. Manage water removed from the well in accordance with 18 AAC 75 or 18 AAC 78.
9. Complete an "as built" drawing/schematic for each constructed monitoring well.
10. Survey wells vertically and horizontally with survey loops that close within 0.01' vertically, and 0.2' horizontally. The well survey data must be provided to ADEC in a written report. Submit a record of the well design, installation, and the materials used to ADEC.

Monitoring Well Development

The primary function of a monitoring well is to provide a representative sample of groundwater as it exists in the formation. The goal of well development is to repair the damage caused during drilling, direct-push emplacement or excavation well installation to the area immediately adjacent to the well, ensuring proper hydraulic connection to the aquifer. Formation changes during well installation are variable, but are usually the compaction of unconsolidated particles surrounding the annulus. In fine-grained soils, this can result in a "mudwall" around the boring annulus, which can impede free flow of the formation water into the well. Development should agitate the adjacent formation and pull fines

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into the well, where they can be removed along with the development water. Well installations in finer-grained deposits are more difficult as the filter pack will not completely stop fines from entering the well.

Common well development methods are a combination of surging, pumping, air or water injection and bailing. In relatively permeable formations, lower a bailer to the water column and surge by use of a surge block attached to tubing to help breakdown any mud wall and prevent particle bridging. Unidirectional flow into the well can cause formation particles to “bridge” together and form blockages. Stopping and starting the pump can aid in a surge toward the formation, which can help break up bridged particles. It is more effective to alternate between using a surge block and bailing or pumping so that there is multidirectional flow on the filter pack around the well. Continue pumping, bailing, and surging until the turbidity decreases. Ideally, the formation water pulled from the well will now be clear. However, it is important not to overdevelop a well by overly aggressive surging. Occasionally, it may not be possible to clear the water from a well due to high concentrations of naturally occurring suspended solids in the aquifer.

Develop groundwater monitoring wells that can be purged dry by first purging the well and then allow the well to refill with formation water. If the recovery rate by the formation water is too slow then add up to one well casing volume of potable water to the well. With water in the well, surge the well vigorously for approximately 10 minutes by using either a surge block or bailer. Add more water as necessary. After surging the well, purge it dry again to complete the development process.

Alternative development procedures may be used if they will not affect the ability of the well to provide representative samples. Wells installed with an annular seal must not be developed until 24 hours after well installation to allow annular seal materials to set or cure. ADEC recognizes that remote site work may make this impractical. Contact your ADEC project manager for site specific approval if development is to be conducted prior to the 24 waiting period. Sample the monitoring well in accordance with the ADEC Draft Field Sampling Guidance.

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ADEC decisions are based on trends over time, not a single sampling event. More than one water sample is required to establish the water quality in any monitoring well, especially a newly installed well. The water quality in a newly installed monitoring well becomes more reliable over time, as the aquifer and the newly installed well reach a state of chemical equilibrium. ASTM standard D5521 (1994) provides guidance on the development of monitoring wells, and standard D5978 (2000) provides guidance on maintaining and repairing a monitoring well. Additionally, EPA (1991) provides a detailed discussion on well development.

- Develop the well by surging, pumping, and bailing.
- Monitor water quality parameters.
- Do not develop the well for at least 24 hours following installation.

1. Measure and record the water level and total depth of the well using a water level indicator.
2. Use a peristaltic pump to remove enough water to measure initial water quality parameters (pH, temperature, conductivity, DO, ORP, and turbidity).
3. Begin well development by surging the monitoring well and removing any sediment from the bottom of the well by slowly lowering a decontaminated surge block into the well so that the surge block is within approximately 0.5 to 1 foot from the bottom of the well. Slowly raise and lower the surge block approximately 1 to 2 feet increments to create a mild surging throughout the well screen.

4. Remove the surge block and immediately begin to pump or bail the water.
5. Repeat this process until accumulated sediment has been removed from the monitoring well.
6. Do not overdevelop the well with overly aggressive surging.
7. Once the water is clear, repeat Steps 4 and 5, continuing to alternate between surging and purging, until measurements stabilize for at least three consecutive water quality parameter readings,according to the following criteria:
    1. ±1.0 degrees Celsius (°C) temperature
    2. ±0.1 pH
    3. ±3 percent conductivity
    4. ±10 millivolts (mV) ORP
    5. ±10 percent DO or 0.2 milligram per liter (mg/L)
    6. ±10 percent turbidity or ≤ 10 nephelometric turbidity units (NTUs)
8. In total, the entire well screen interval should be developed and surging should be conducted for a minimum of 10 minutes.
9. If the well is purged dry at any point during development, approximately one well casing of clean, potable water can be introduced into the well and surging can continue. After surging, purge the well dry again to complete the development process by removing at least the amount of potable water added to the well. If the well will recover naturally, continue development with formation water only.
10. The well will be considered adequately developed and development can stop after the water quality parameters have stabilized or a minimum of 10 well casing volumes (see Attachment 1) have been removed from the well (ADEC, 1992).
11. Measure and record a final depth to water and total well depth measurement after well development.
12. Record all well development data in a field notebook.

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Well Purging

Well purging is the process of removing stagnant water from a monitoring well prior to sampling, causing it to be replaced by groundwater from the adjacent formation. Prior to purging, three measurements need to be recorded: the inside diameter of the well, the depth to water in the well, and the depth to the bottom of the well. With that information, the volume of the water in the well casing needs to be calculated and recorded.

When purging monitoring wells prior to sampling:

- CES will remove at least three casing volumes,
- monitor water quality parameters until a minimum of three (minimum of four if using temperature as an indicator) of the parameters listed below stabilize, or
- for low yield wells, the entire well casing is evacuated.

Water quality parameters are considered stable when three successive readings, collected 3-5 minutes apart, are within:

- ± 3% for temperature (minimum of ± 0.2°C)
- ± 0.1 for pH
- ± 3% for conductivity
- ± 10 mv for redox potential
- ± 10% for dissolved oxygen (DO), and
- ± 10% for turbidity

A minimum of three (minimum of four if using temperature as an indicator) of these parameters must be monitored and recorded. Low flow purging and sampling are particularly useful for wells that purge dry or take one hour or longer to recover. If a well is low yield and purged dry, do not collect a sample until it has recharged to approximately 80% of its pre-purge volume, when practical.

General Procedures for Measuring Groundwater Quality Parameters

1. Calibrate instruments according to the manufacturer's procedures.
2. Secure the meter to the flow-through cell.
3. Connect a short discharge tube to the effluent connector and run the other end of the discharge tube into a 5-gallon purge water capture bucket.
4. Start purging at a flow rate of approximately 1 liter (0.25 gallon) every 3 minutes or 0.1 gallon per minute (gal/min).
5. Measure and record the groundwater parameters and current groundwater level until the parameters stabilize according to the following stabilization criteria, or until three well casing volumes are purged.
6. Groundwater parameters are considered stable after purging if three successive readings are within:
    - ± 1.0 °C temperature
    - ± 0.1 pH
    - ± 3 percent conductivity
    - ± 10 millivolt (mV) ORP
    - ± 10 percent parts per million (ppm) DO or 0.2 milligrams per liter (mg/L)
    - ± 10 percent turbidity or ≤ 10 NTUs

    - • ORP and DO measurements should correlate with each other. Generally ORP should be negative whenever DO is near or less than 1 mg/L; likewise, DO should be greater than 1 mg/L if ORP is positive.
    - • DO measurements should be positive and range between 0 and 14.62 mg/L.
    - ORP measurements should range between -500 and 275 mV.
    - pH of environmental samples will typically range from 6 to 8 pH units.

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Prior to sampling, determine depth to groundwater to within 0.01 feet. Check the monitoring well for the presence of NAPL that might be floating on top of the water or in a separate layer at the bottom of the casing. If wells contain NAPL then alternate wells that are representative of the affected groundwater should be sampled, if available. Alternatively, water samples should be collected using methods that minimize the potential for NAPL inclusion in samples that will be analyzed to measure dissolved phase concentrations; the field notes and report must describe the fact that NAPL was present and the observed thickness of the NAPL.

Identify NAPL by an electronic device designed to detect non-aqueous liquids and to measure the thickness of the non-aqueous layer. Because of the lower density of the NAPL, bailers will measure a smaller NAPL thickness than is actually in the monitoring well, or measure no NAPL at all.

Depth to Water and Depth to Product Measurement

1. Slowly lower the water level meter or oil-water indicator probe down the monitoring well until the probe contacts the groundwater or NAPL surface, as indicated by the audible alarm.
2. Raise the probe out of the water or NAPL until the audible alarm stops. Continue raising and lowering the probe until a precise level is determined within 0.01 foot.

3. If NAPL is present in the well, measure and record the depth from the TOC reference point to the top surface of the NAPL layer (that is, DTP). The oil-water indicator probe alarm will sound a continuous tone when NAPL is detected.
4. Continue to lower the probe until the meter indicates the presence of groundwater. The alarm will typically emit a beep when water is detected. Measure the first static groundwater level and record the measurement (DTW) from the reference point to the top of the static groundwater level.
5. Record the measurements in the field log book.

Total Well Depth Measurement

1. Slowly lower the water level meter until the cable goes slack.
2. Gently raise and lower the water level meter probe to tap the bottom of the well.
3. Record the reading on the cable at the established reference point to the nearest 0.01 foot.

Groundwater Well Sampling

1. Briefly turn off the pump and disconnect the aboveground end of the pump intake tube from water quality meter and flow-through cell before sampling. Do not sample from the discharge of the flow-through-cell.
2. Turn the pump back on and continue to pump at a flow rate of approximately 0.1 gal/min. Re-verify the pumping rate does not lower the water level in the well by more than 0.3 foot.
3. Fill the laboratory-supplied analytical sample containers in the order of volatility.
4. Preserve containers immediately if preservative is not already in the containers, and unless specified otherwise, at a minimum, immediately cool the samples to 4 ±2°C and maintain this temperature through delivery to laboratory until the samples are analyzed.
5. Record the sample ID, date and time, sampler, analytes, and other sample information on the sample bottle labels, the sample chain-of-custody, and field log book.

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Designation: D 5092 – 04$^{\epsilon 1}$

Standard Practice for Design and Installation of Ground Water Monitoring Wells[1]

This standard is issued under the fixed designation D 5092; 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 ( ) indicates an editorial change since the last revision or reapproval.

[1] NOTE—Editorial changes were made throughout in June 2004.

1. Scope

1.1 This practice describes a methodology for designing and installing conventional (screened and filter-packed) ground-water monitoring wells suitable for formations ranging from unconsolidated aquifers (i.e., sands and gravels) to granular materials having grain-size distributions with up to 50 % passing a #200 sieve and as much as 20 % clay-sized material (i.e., silty fine sands with some clay). Formations finer than this (i.e., silts, clays, silty clays, clayey silts) should not be monitored using conventional monitoring wells, as representative ground-water samples, free of artifactual turbidity, cannot be assured using currently available technology. Alternative monitoring technologies (not described in this practice) should be used in these formations

1.2 The recommended monitoring well design and installation procedures presented in this practice are based on the assumption that the objectives of the program are to obtain representative ground-water samples and other representative ground-water data from a targeted zone of interest in the subsurface defined by site characterization.

1.3 This practice, in combination with proper well development (D 5521), proper ground-water sampling procedures (D 4448), and proper well maintenance and rehabilitation (D 5978), will permit acquisition of ground-water samples free of artifactual turbidity, eliminate siltation of wells between sampling events, and permit acquisition of accurate ground-water levels and hydraulic conductivity test data from the zone screened by the well. For wells installed in fine-grained formation materials (up to 50 % passing a #200 sieve), it is generally necessary to use low-flow purging and sampling techniques (D 6771) in combination with proper well design to collect turbidity-free samples.

1.4 This practice applies primarily to well design and installation methods used in drilled boreholes. Other Standards, including Guide D 6724 and Practice D 6725, cover installation of monitoring wells using direct-push methods.

1.5 The values stated in inch-pound units are to be regarded as standard. The values in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, 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.*

1.7 *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. Nat 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.*

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2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 150 Specification for Portland Cement

C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates

D 421 Practice for Dry Preparation of Soil Samples for Particle Size Analysis and Determination of Soil Constants

D 422 Test Method for Particle Size Analysis of Soils

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 1452 Practice for Soil Investigation and Sampling by Auger Borings

D 1586 Method for Penetration Test and Split-Barrel Sampling of Soils

D 1587 Practice for Thin-Walled Tube Sampling of Soils

D 2113 Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

D 2217 Practice for Wet Preparation of Soil Samples for Particle Size Analysis and Determination of Soil Constants

[1] This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21.05 on Design and Installation of Ground-Water Monitoring Wells.

Current edition approved Jan. 1, 2004. Published March 2004. Originally approved in 1990. Last previous edition approved in 2002 as D 5092 – 02.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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D 2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)
D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)
D 3282 Practice for Classification of Soils and Soil Aggregate Mixtures for Highway Construction Purposes
D 3441 Test Method for Deep, Quasi-Static, Cone and Friction Cone Penetration Tests of Soil
D 3550 Practice for Ring Lined Barrel Sampling of Soils
D 4220 Practice for Preserving and Transporting Soil Samples
D 4700 Guide for Soil Sampling from the Vadose Zone
D 4750 Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well)
D 5079 Practices for Preserving and Transporting Rock Core Samples
D 5088 Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites
D 5254 Practice for Minimum Set of Data Elements to Identify a Ground-Water Site
D 5299 Guide for Decommissioning of Ground-Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities
D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock
D 5518 Guide for Acquisition of File Aerial Photography and Imagery for Establishing Historic Site Use and Surficial Conditions
D 5521 Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers
D 5608 Practice for Decontamination of Field Equipment Used at Low-Level Radioactive Waste Sites
D 5730 Guide to Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone, and Ground Water
D 5753 Guide for Planning and Conducting Borehole Geophysical Logging
D 5777 Guide for Using the Seismic Refraction Method for Subsurface Investigation
D 5781 Guide for Use of Dual-Wall Reverse-Circulation Drilling for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5782 Guide for Use of Direct Air-Rotary Drilling for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5784 Guide for Use of Hollow Stem Augers for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5787 Practice for Monitoring Well Protection
D 5872 Guide for the Use of Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5875 Guide for the Use of Cable Tool Drilling and Sampling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
D 5876 Guide for the Use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
D 5978 Guide for Maintenance and Rehabilitation of Ground-Water Monitoring Wells
D 5979 Guide for Conceptualization and Characterization of Ground-Water Systems
D 6001 Guide for Direct-Push Water Sampling for Geoenvironmental Investigations
D 6067 Guide for Using the Electronic Cone Penetrometer for Environmental Site Characterization
D 6167 Guide for Conducting Borehole Geophysical Logging
D 6169 Guide to the Selection of Soil and Rock Sampling Devices Used With Drilling Rigs for Environmental Investigations
D 6235 Practice for Expedited Site Characterization of Vadose Zone and Ground-Water Contamination at Hazardous Waste Contaminated Sites
D 6274 Guide for Conducting Borehole Geophysical Logging—Gamma
D 6282 Guide for Direct-Push Soil Sampling for Environmental Site Characterization
D 6286 Guide to the Selection of Drilling Methods for Environmental Site Characterization
D 6429 Guide for Selecting Surface Geophysical Methods
D 6430 Guide for Using the Gravity Method for Subsurface Investigation
D 6431 Guide for Using the Direct Current Resistivity Method for Subsurface Investigation
D 6432 Guide for Using the Surface Ground Penetrating Radar Method for Subsurface Investigation
D 6519 Practice for Sampling of Soil Using the Hydraulically Operated Stationary Piston Sampler
D 6639 Guide for Using the Frequency Domain Electromagnetic Method for Subsurface Investigations
D 6640 Guide for Collection and Handling of Soils Obtained in Core Barrel Samplers for Environmental Investigations
D 6724 Guide for the Installation of Direct-Push Ground-Water Monitoring Wells
D 6725 Practice for the Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers
D 6771 Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations
F 480 Specification for Thermoplastic Well Casing and Couplings Made in Standard Dimension Ratios (SDR), Schedule 40 and Schedule 80

3. Terminology

3.1 *Definitions:*

3.1.1 *annular space; annulus*—the space between two concentric strings of casing, or between the casing and the

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borehole wall. This includes the space(s) between multiple strings of casing in a borehole installed either concentrically or adjacent to one another.

3.1.2 *artifactual turbidity*—particulate matter that is not naturally mobile in the ground-water system and that is produced in some way by the ground-water sampling process. May consist of particles introduced to the subsurface during drilling or well construction, sheared from the target monitoring zone during pumping or bailing the well, or produced by exposure of ground water to atmospheric conditions.

3.1.3 *assessment monitoring*—an investigative monitoring program that is initiated after the presence of a contaminant in ground water has been detected. The objective of this program is to determine the concentration of constituents that have contaminated the ground water and to quantify the rate and extent of migration of these constituents.

3.1.4 *ballast*—materials used to provide stability to a buoyant object (such as casing within a water-filled borehole).

3.1.5 *borehole*—an open or uncased subsurface hole, generally circular in plan view, created by drilling.

3.1.6 *borehole log*—the record of geologic units penetrated, drilling progress, depth, water level, sample recovery, volumes, and types of materials used, and other significant facts regarding the drilling and/or installation of an exploratory borehole or well.

3.1.7 *bridge*—an obstruction within the annulus that may prevent circulation or proper placement of annular fill materials.

3.1.8 *casing*—pipe, finished in sections with either threaded connections or beveled edges to be field welded, which is installed temporarily or permanently either to counteract caving, to advance the borehole, or to isolate the zone being monitored, or any combination of these.

3.1.9 *casing, protective*—a section of larger diameter pipe that is placed over the upper end of a smaller diameter monitoring well riser or casing to provide structural protection to the well, to prevent damage to the well, and to restrict unauthorized access into the well.

3.1.10 *casing, surface*—pipe used to stabilize a borehole near the surface during the drilling of a borehole that may be left in place or removed once drilling is completed.

3.1.11 *caving; sloughing*—the inflow of unconsolidated material into a borehole that occurs when the borehole walls lose their cohesiveness.

3.1.12 *cement*—commonly known as Portland cement. A mixture that consists of calcareous, argillaceous, or other silica-, alumina-, and iron-oxide-bearing materials that is manufactured and formulated to produce various types which are defined in Specification C 150. Portland cement is considered a hydraulic cement because it must be mixed with water to form a cement-water paste that has the ability to harden and develop strength even if cured under water.

3.1.13 *centralizer*—a device that assists in the centering of a casing or riser within a borehole or another casing.

3.1.14 *confining unit*—a body of relatively low hydraulic conductivity formation material stratigraphically adjacent to one or more aquifers. Synonymous with "aquiclude,"" aquitard," and "aquifuge."

3.1.15 *detection monitoring*—a program of monitoring for the express purpose of determining whether or not there has been a contaminant release to ground water.

3.1.16 *d-10*—the diameter of a soil particle (preferably in mm) at which 10 % by weight (dry) of the particles of a particular sample are finer. Synonymous with the effective size or effective grain size.

3.1.17 *d-60*—the diameter of a soil particle (preferably in mm) at which 60 % by weight (dry) of the particles of a particular sample are finer.

3.1.18 *flush joint or flush coupled*—casing or riser with ends threaded such that a consistent inside and outside diameter is maintained across the threaded joints or couplings.

3.1.19 *gravel pack*—common term used to refer to the primary filter pack of a well (see *primary filter pack*).

3.1.20 *grout (monitoring wells)*—a low-permeability material placed in the annulus between the well casing or riser and the borehole wall (in a single-cased monitoring well), or between the riser and casing (in a multi-cased monitoring well), to prevent movement of ground water or surface water within the annular space.

3.1.21 *hydrologic unit*—geologic strata that can be distinguished on the basis of capacity to yield and transmit fluids. Aquifers and confining units are types of hydrologic units. Boundaries of a hydrologic unit may not necessarily correspond either laterally or vertically to lithostratigraphic formations.

3.1.22 *multi-cased well*—a well constructed by using successively smaller diameter casings with depth.

3.1.23 *neat cement*—a mixture of Portland cement (Specification C 150) and water.

3.1.24 *packer (monitoring wells)*—a transient or dedicated device placed in a well that isolates or seals a portion of the well, annulus, or borehole at a specific level.

3.1.25 *piezometer*—a small-diameter well with a very short screen that is used to measure changes in hydraulic head, usually in response to pumping a nearby well. Synonymous with observation well.

3.1.26 *primary filter pack*—a clean silica sand or sand and gravel mixture of selected grain size and gradation that is installed in the annular space between the borehole wall and the well screen, extending an appropriate distance above the screen, for the purpose of retaining and stabilizing the particles from the adjacent formation(s). The term is used in place of *gravel pack*.

3.1.27 *PTFE tape*—joint sealing tape composed of polytetrafluoroethylene.

3.1.28 *riser*—the pipe or well casing extending from the well screen to just above or below the ground surface.

3.1.29 *secondary filter pack*—a clean, uniformly graded sand that is placed in the annulus between the primary filter pack and the overlying seal, or between the seal and overlying grout backfill, or both, to prevent intrusion of the seal or grout, or both, into the primary filter pack.

3.1.30 *sediment sump*—a blank extension of pipe or well casing, closed at the bottom, beneath the well screen used to collect fine-grained material from the filter pack and adjacent

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formation materials during the process of well development. Synonymous with rat trap or tail pipe.

3.1.31 *single-cased well*—a monitoring well constructed with a riser but without an exterior casing.

3.1.32 *static water level*—the elevation of the top of a column of water in a monitoring well or piezometer that is not influenced by pumping or conditions related to well installation, or hydraulic testing.

3.1.33 *tamper*—a heavy cylindrical metal section of tubing that is operated on a wire rope or cable. It either slips over the riser and fits inside the casing or borehole annulus, or fits between the riser and annulus. It is generally used to tamp annular sealants or filter pack materials into place and to prevent bridging or break bridges that form in the annular space.

3.1.34 *target monitoring zone*—the ground-water flow path from a particular area or facility in which monitoring wells will be screened. The target monitoring zone should be an interval in subsurface materials in which there is a reasonable expectation that a monitoring well will intercept ground water moving beneath an area or facility and any migrating contaminants that may be present.

3.1.35 *tremie pipe*—a small-diameter pipe or tube that is used to transport filter pack materials and annular seal materials from the ground surface into an annular space.

3.1.36 *uniformity coefficient*—the ratio of d-60/d-10, where d-60 and d-10 are particle diameters corresponding to 60 % and 10 % finer on the cumulative particle size curve, respectively.

3.1.37 *uniformly graded*—a quantitative definition of the particle size distribution of a soil that consists of a majority of particles being of approximately the same diameter. A granular material is considered uniformly graded when the uniformity coefficient is less than about five (Test Method D 2487). Comparable to the geologic term *well sorted*.

3.1.38 *vented cap*—a cap with a small hole that is installed on top of the riser.

3.1.39 *weep hole*—a small-diameter hole (usually ¼ in.) drilled into the protective casing above the ground surface that serves to drain out water that may enter the annulus between the riser and the protective casing.

3.1.40 *well completion diagram*—a record that illustrates the details of a well installation.

3.1.41 *well screen*—a device used to retain the primary or natural filter pack; usually a cylindrical pipe with openings of a uniform width, orientation, and spacing.

4. Significance and Use

4.1 This practice for the design and installation of ground-water monitoring wells will promote *(1)* efficient and effective site hydrogeological characterization; *(2)* durable and reliable well construction; and *(3)* acquisition of representative ground-water quality samples, ground-water levels, and hydraulic conductivity testing data from monitoring wells. The practices established herein are affected by governmental regulations and by site-specific geological, hydrogeological, climatological, topographical, and subsurface geochemical conditions. To meet these geoenvironmental challenges, this practice promotes the development of a conceptual hydrogeologic model prior to monitoring well design and installation.

4.2 A properly designed and installed ground water monitoring well provides essential information on one or more of the following subjects:

4.2.1 Formation geologic and hydraulic properties;

4.2.2 Potentiometric surface of a particular hydrologic unit(s);

4.2.3 Water quality with respect to various indicator parameters; and

4.2.4 Water chemistry with respect to a contaminant release.

5. Site Characterization

5.1 *General*—A thorough knowledge of site-specific geologic, hydrologic and geochemical conditions is necessary to properly apply the monitoring well design and installation procedures contained within this practice. Development of a conceptual site model, that identifies potential flow paths and the target monitoring zone(s), and generates a 3-D picture of contaminant distribution and contaminant movement pathways, is recommended prior to monitoring well design and installation. Development of the conceptual site model is accomplished in two phases -- an initial reconnaissance, after which a preliminary conceptual model is created, and a field investigation, after which a revised conceptual model is formulated. When the hydrogeology of a project area is relatively uncomplicated and well documented in the literature, the initial reconnaissance may provide sufficient information to identify flow paths and the target monitoring zone(s). However, where limited or no background data are available or where the geology is complex, a field investigation will be required to develop the necessary conceptual site model.

5.2 *Initial Reconnaissance of Project Area*—The goal of the initial reconnaissance of the project area is to identify and locate those zones or preferential flow pathways with the greatest potential to transmit fluids from the project area. Identifying these flow pathways is the first step in selecting the target ground-water monitoring zone(s).

5.2.1 *Literature Search*—Every effort should be made to collect and review all applicable field and laboratory data from previous investigations of the project area. Information such as, but not limited to, topographic maps, aerial imagery (see Guide D 5518), site ownership and utilization records, geologic and hydrogeologic maps and reports, mineral resource surveys, water well logs, information from local well drillers, agricultural soil reports, geotechnical engineering reports, and other engineering maps and reports related to the project area should be reviewed to locate relevant site information.

5.2.2 *Field Reconnaissance*—Early in the investigation, the soil and rocks in open cut areas (e.g., roadcuts, streamcuts) in the vicinity of the project should be studied, and various soil and rock profiles noted. Special consideration should be given to soil color and textural changes, landslides, seeps, and springs within or near the project area.

5.2.3 *Preliminary Conceptual Model*—The distribution of the predominant soil and rock units likely to be found during subsurface exploration may be hypothesized at this time in a preliminary conceptual site model using information obtained in the literature search and field reconnaissance. In areas where

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the geology is relatively uniform, well documented in the literature, and substantiated by the field reconnaissance, further refinement of the conceptual model may not be necessary unless anomalies are discovered in the well drilling stage.

5.3 *Field Investigation*—The goal of the field investigation is to refine the preliminary conceptual site model so that the target monitoring zone(s) is (are) identified prior to monitoring well installation.

5.3.1 *Exploratory Borings and Direct-Push Methods*—Characterization of the flow paths conceptualized in the initial reconnaissance involves defining the porosity (type and amount), hydraulic conductivity, stratigraphy, lithology, gradation and structure of each hydrologic unit encountered beneath the site. These characteristics are defined by conducting an exploratory program which may include drilled soil borings (see Guide D 6286 for selection of drilling methods) and direct-push methods (e.g., cone penetrometers [see Test Method D 3441 or Guide D 6067] or direct-push machines using soil sampling, ground-water sampling and/or electrical conductivity measurement tools [see Guides D 6282 and D 6001]). Exploratory soil borings and direct-push holes should be deep enough to develop the required engineering and hydrogeologic data for determining the preferential flow pathway(s), target monitoring zone(s), or both.

5.3.1.1 *Sampling*—Soil and rock properties should not be predicted wholly on field description or classification, but should be confirmed by laboratory and/or field tests made on samples or in boreholes or wells. Representative soil or rock samples of each material that is significant to the design of the monitoring well system should be obtained and evaluated by a geologist, hydrogeologist, soil scientist or engineer trained and experienced in soil and rock analysis. Soil sample collection should be conducted according to Practice D 1452, Test Method D 1586, Practice D 3550, Practice D 6519 or Practice D 1587, whichever is appropriate given the anticipated characteristics of the soil samples (see Guide D 6169 for selection of soil sampling methods). Rock samples should be collected according to Practice D 2113. Soil samples obtained for evaluation of hydraulic properties should be containerized and identified for shipment to a laboratory. Special measures to preserve either the continuity of the sample or the natural moisture are not usually required. However, soil and rock samples obtained for evaluation of chemical properties often require special field preparation and preservation to prevent significant alteration of the chemical constituents during transportation to a laboratory (see Practice D 6640). Rock samples for evaluation of hydraulic properties are usually obtained using a split-inner-tube core barrel. Evaluation and logging of the core samples is usually done in the field before the core is removed from the core barrel.

5.3.1.2 *Boring Logs*—Care should be taken to prepare and retain a complete boring log and sampling record for each exploratory soil boring or direct-push hole (see Guide D 5434).

NOTE 1—Site investigations conducted for the purpose of generating data for the installation of ground-water monitoring wells can vary greatly due to the availability of reliable site data or the lack thereof. The general procedure would be as follows: *(1)* gather factual data regarding the surficial and subsurface conditions, *(2)* analyze the data, *(3)* develop a conceptual model of the site conditions, *(4)* locate the monitoring wells based on the first three steps. Monitoring wells should only be installed with sufficient understanding of the geologic, and hydrologic and geochemical conditions present at the site. Monitoring wells often serve as part of an overall site investigation for a specific purpose, such as determining the extent of contamination present, or for predicting the effectiveness of aquifer remediation. In these cases, extensive additional geotechnical and hydrogeologic information may be required that would go beyond the Section 5 Site Characterization description.

Boring logs should include the location, geotechnical data (that is, penetration rates or blow counts), and sample description information for each material identified in the borehole either by symbol or word description, or both. Description and identification of soils should be in accordance with Practice D 2488; classification of soils should be in accordance with either Practice D 2487 or Practice D 3282. Identification of rock material should be based on Nomenclature C 294 or by an appropriate geologic classification system. Observations of seepage, free water, and water levels should also be noted. The boring logs should be accompanied by a report that includes a description of the area investigated; a map illustrating the vertical and horizontal location (with reference to either North American Vertical Datum of 1988 [NAVD 88] or to a standardized survey grid) of each exploratory soil boring or test pit, or both; and color photographs of rock cores, soil samples, and exposed strata labeled with a date and identification.

5.3.2 *Geophysical Exploration*—Geophysical surveys may be used to supplement soil boring and outcrop observation data and to aid in interpretation between soil borings. Appropriate surface and borehole geophysical methods for meeting site-specific project objectives can be selected by consulting Guides D 6429 and D 5753 respectively. Surface geophysical methods such as seismic (Guide D 5777), electrical-resistivity (Guide D 6431), ground-penetrating radar (Guide D 6432), gravity (Guide D 6430) and electromagnetic conductance surveys (Guide D 6639) can be particularly valuable when distinct differences in the properties of contiguous subsurface materials are indicated. Borehole methods such as resistivity, gamma, gamma-gamma, neutron, and caliper logs (see Guide D 6167) can be useful to confirm specific subsurface geologic conditions. Gamma logs (Guide D 6274) are particularly useful in existing cased wells.

5.3.3 *Ground-Water Flow Direction*—Ground-water flow direction is generally determined by measuring the vertical and horizontal hydraulic gradient within each conceptualized flow pathway. However, because water will flow along the pathways of least resistance (within the highest hydraulic conductivity formation materials at the site), actual flow direction may be oblique to the hydraulic gradient (within buried stream channels or glacial valleys, for example). Flow direction is determined by first installing piezometers in the exploratory soil borings that penetrate the zone(s) of interest at the site. The depth and location of the piezometers will depend upon anticipated hydraulic connections between conceptualized flow pathways and their respective lateral direction of flow. Following careful evaluation, it may be possible to utilize existing private or public wells to obtain water-level data. The construction integrity of such wells should be verified to ensure that the water levels obtained from the wells are representative only of the zone(s) of interest. Following water-level data acquisition,

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a potentiometric surface map should be prepared. Flow pathways are ordinarily determined to be at right angles, or nearly so, to the equipotential lines, though consideration of complex geology can result in more complex interpretations of flow

5.4 *Completing the Conceptual Model*—A series of geologic and hydrogeologic cross sections should be developed to refine the conceptual model. This is accomplished by first plotting logs of soil and rock observed in the exploratory soil borings or test pits, and interpreting between these logs using the geologic and engineering interrelationships between other soil and rock data observed in the initial reconnaissance or with geophysical techniques. Extrapolation of data into adjacent areas should be done only where geologically uniform subsurface conditions are known to exist. The next step is to integrate the geologic profile data with the potentiometric data for both vertical and horizontal hydraulic gradients. Plan view and cross-sectional flow nets should be constructed. Following the analysis of these data, conclusions can be made as to which flow pathway(s) is (are) the appropriate target monitoring zone(s).

NOTE 2—UUse of ground-water monitoring wells is difficult and may not be a reliable technology in fine-grained, low hydraulic conductivity formation materials with primary porosity because of *(1)* the disproportionate influence that microstratigraphy has on ground-water flow in fine-grained strata; *(2)* the proportionally higher vertical flow component in low hydraulic conductivity strata; and *(3)* the presence of indigenous metallic and inorganic constituents in the matrix that make water-quality data evaluation difficult.

6. Monitoring Well Construction Materials

6.1 *General*—The materials that are used in the construction of a monitoring well that come in contact with water samples should not alter the chemical quality of the sample for the constituents being examined. The riser, well screen, and annular seal installation equipment should be cleaned immediately prior to well installation (see either Practice D 5088 or D 5608) or certified clean from the manufacturer and delivered to the site in a protective wrapping. Samples of the riser and screen material, cleaning water, filter pack, annular seal, bentonite, and mixed grout should be retained to serve as quality control until the completion of at least one round of ground- water quality sampling and analysis has been completed.

6.2 *Water*—Water used in the drilling process, to prepare grout mixtures and to decontaminate the well screen, riser, and annular sealant injection equipment, should be obtained from a source of known chemistry that does not contain constituents that could compromise the integrity of the well installation.

6.3 *Primary Filter Pack*:

6.3.1 *General*—The purposes of the primary filter pack are to act as a filter that retains formation material while allowing ground water to enter the well, and to stabilize the formation to keep it from collapsing on the well. The design of the primary filter pack is based on the grain-size distribution of the formation material (as determined by sieve analysis—see Test Method D 422) to be retained. The grain size distribution of the primary filter pack must be fine enough to retain the formation, but coarse enough to allow for unrestricted movement of ground water into and through the monitoring well. The design of the well screen (see 6.4.3) must be done in concert with the design of the filter pack. After development, a monitoring well with a correctly designed and installed filter pack and screen combination should produce samples free of artifactual turbidity.

6.3.2 *Materials*—The primary filter pack should consist of an inert granular material (generally ranging from gravel to very fine sand, depending on formation grain size distribution) of selected grain size and gradation that is installed in the annulus between the well screen and the borehole wall. Washed and screened silica sands and gravels, with less than 5 % non-siliceous materials, should be specified.

6.3.3 *Design*—The design theory of filter pack gradation is based on mechanical retention of formation materials.

6.3.3.1 1 For formation materials that are relatively coarse-grained (i.e., fine, medium and coarse sands and gravels), the grain size distribution of the primary filter pack is determined by calculating the d-30 (30 % finer) size, the d-60 (60 % finer) size, and the d-10 (10 % finer) size of the filter pack. The first point on the filter pack grain-size distribution curve is the d-30 size. The primary filter pack is usually selected to have a d-30 grain size that is about 4 to 6 times greater than the d-30 grain size of the formation material being retained (see Fig. 1). A multiplication factor of 4 is used if the formation material is relatively fine-grained and well sorted or uniform (small range in grain sizes); a multiplication factor of 6 is used if the formation is relatively coarse grained and poorly sorted or non-uniform (large range in grain sizes). Thus, 70 % of the filter pack will have a grain size that is 4 to 6 times larger than the d-30 size of the formation materials. This ensures that the filter pack is coarser (with a higher hydraulic conductivity) than the formation material, and allows for unrestricted ground-water flow from the formation into the monitoring well.

The next 2 points on the filter pack grain-size distribution curve are the d-60 and d-10 grain sizes. These are chosen so that the ratio between the two grain sizes (the uniformity coefficient) is less than 2.5. This ensures that the filter pack has a small range in grain sizes and is uniform (see technical Note 5). The d-60 and d-10 grain sizes of the filter pack are calculated by a trial and error method using grain sizes that are close to the d-30 size of the filter pack. After the d-30, d-60 and d-10 sizes of the filter pack are determined, a smooth curve is drawn through these points. The final step in filter pack design is to specify the limits of the grain size envelope, which defines the permissible range in grain sizes for the filter pack. The permissible range on either side of the grain size curve is 8 %. The boundaries of the grain size envelope are drawn on either side of the filter pack grain-size distribution curve, and filter pack design is complete. A filter medium having a grain-size distribution as close as possible to this curve is then obtained from a local sand supplier.

6.3.3.2 In formation materials that are predominantly fine-grained (finer than fine to very fine sands), soil piping can occur when a hydraulic gradient exists between the formation and the well (as would be the case during well development and sampling). To prevent soil piping in these materials, the following criteria are used for designing granular filter packs:

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d-15 of filter / d-85 of formation </= 4 to 5 and d-15 of filter / d-15 of formation >/= 4 to 5

The left half of this equation is the fundamental criterion for the prevention of soil piping through a granular filter, while the right half of the equation is the hydraulic conductivity criterion. This latter criterion serves the same purpose as multiplying the d-30 grain size of the formation by a factor of between 4 and 6 for coarser formation materials. Filter pack materials suitable for retaining formation materials in formations that are predominantly fine-grained are themselves, by necessity, relatively fine-grained (e.g., fine to very fine sands), presenting several problems for well designers and installers. First, well screen slot sizes suitable for retaining such fine-grained filter pack materials are not widely available (the smallest commercially available slotted well casing is 0.006 in. [6 slot]; the smallest commercially available continuous-slot wire-wound screen is 0.004 in. [4 slot]). Second, the finest filter pack material practical for conventional (tremie tube) installation is a 40 by 70 (0.008 by 0.018 in.) sand, which can be used with a well screen slot as small as 0.008-in. (8 slot). Finer grained filter pack materials cannot be placed practically by either tremie tubes or pouring down the annular space or down augers. Thus, the best method for ensuring proper installation of filter packs in predominantly fine-grained formation materials is to use pre-packed or sleeved screens, which are described in detail in Practice D 6725. A 50 by 100 (0.011 by 0.006 in.) filter-pack sand can be used with a 0.006-in. slot size pre-packed or sleeved screen, and a 60 by 120 (0.0097 by 0.0045 in.) filter-pack sand can be used with a 0.004-in. (4 slot) slot size pre-packed or sleeved screen. Filter packs that are finer than these (e.g., sands as fine as 100 by 120 [0.006 by 0.0045 in.], or silica flour as fine as 200 mesh [0.003 in.]) can only be installed within stainless steel mesh sleeves that can be placed over pipe-based screens. While these sleeves, or the space between internal and external screens in a pre-packed well screen may be as thin as 1/2-in. (1.27 cm), the basis for mechanical retention dictates that a filter-pack thickness of only two or three grain diameters is needed to contain and control formation materials. Laboratory tests have demonstrated that a properly sized filter pack material with a thickness of less than 1/2-in. (1.27 cm) successfully retains formation particles regardless of the velocity of water passing through the filter pack [3].

[3] (1) Driscoll, F.G., 1986, Groundwater and Wells, Johnson Division, St. Paul, MN, pg.443

6.3.3.3 The limit of mechanical filtration for monitoring wells is defined by the finest filter pack material that can be practically installed via a pre-packed or sleeved screen—silica flour with a grain size of 0.003 in. (200 mesh), encased within a very fine mesh screen of stainless steel or other suitable material. This fine a filter pack material will retain formation material as fine as silt, but not clay. Formations with a small fraction of clay (up to about 20 %) can be successfully monitored, as long as the wells installed in these formations are properly developed (see Guide D 5521). For mechanical filtration to be effective in formations with more than 50 % fines, the filter pack design would have to include silt-sized particles in the filter pack in order to meet the design criteria, which is impractical, as placement would be impossible and screen mesh fine enough to retain the material is not commercially available. Therefore, formations with more than 50 % passing a #200 sieve, and having more than 20 % clay-sized material, should not be monitored using conventional well designs. Alternative monitoring technologies should be used in these formations..

NOTE 3—When installing a monitoring well in solution-channeled limestone or highly fractured bedrock, the borehole configuration of void spaces within the formation surrounding the borehole is often unknown. Therefore, the installation of a filter pack becomes difficult and may not be possible.

NOTE 4—This practice presents a design for monitoring wells that will be effective in the majority of formations. Applicable state guidance may differ from the designs contained in this practice.

NOTE 5—Because the well screen slots have uniform openings, the filter pack should be composed of particles that are as uniform in size as is practical. Ideally, the uniformity coefficient (the quotient of the 60 % passing, D-60 size divided by the 10 % passing D-10 size [effective size]) of the filter pack should be 1.0 (that is, the D-60 % and the D-10 % sizes should be identical). However, a more practical and consistently achievable uniformity coefficient for all ranges of filter pack sizes is 2.5. This value of 2.5 should represent a maximum value, not an ideal.

NOTE 6—Although not recommended as standard practice, often a project requires drilling and installing the well in one phase of work. Therefore, the filter pack materials must be ordered and delivered to the drill site before soil samples can be collected. In these cases, the suggested well screen slot size and filter pack material combinations are presented in Table 1.

NOTE 7—Silica flour can alter water chemistry, particularly for transuranics, and its use should be evaluated against the monitoring program analytes

6.4 *Well Screen*:

6.4.1 *General*—The purposes of the well screen are to provide designed openings for ground-water flow through the well, and to prevent migration of filter pack and formation

TABLE 1 Recommended (Achievable) Filter Pack Characteristics for Common Screen Slot Sizes

Size of Screen Opening, mm (in.)	Slot No.	Sand Pack Mesh Size Name(s)	1 % Passing Size (D-1), mm	Effective Size, (D-10), mm	30 % Passing Size (D-30), mm	Range of Uniformity Coefficient	Roundness (Powers Scale)
0.125 (0.005)	5[A]	100	0.09 to 0.12	0.14 to 0.17	0.17 to 0.21	1.3 to 2.0	2 to 5
0.25 (0.010)	10	20 to 40	0.25 to 0.35	0.4 to 0.5	0.5 to 0.6	1.1 to 1.6	3 to 5
0.50 (0.020)	20	10 to 20	0.7 to 0.9	1.0 to 1.2	1.2 to 1.5	1.1 to 1.6	3 to 6
0.75 (0.030)	30	10 to 20	0.7 to 0.9	1.0 to 1.2	1.2 to 1.5	1.1 to 1.6	3 to 6
1.0 (0.040)	40	8 to 12	1.2 to 1.4	1.6 to 1.8	1.7 to 2.0	1.1 to 1.6	4 to 6
1.5 (0.060)	60	6 to 9	1.5 to 1.8	2.3 to 2.8	2.5 to 3.0	1.1 to 1.7	4 to 6
2.0 (0.080)	80	4 to 8	2.0 to 2.4	2.4 to 3.0	2.6 to 3.1	1.1 to 1.7	4 to 6

[A] A 5-slot (0.152-mm) opening is not currently available in slotted PVC but is available in Vee wire PVC and Stainless; 6-slot opening may be substituted in these cases.

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material into the well. The well screen design is based on either the grain-size distribution of the formation (in the case of a well with a naturally developed filter pack), or the grain-size distribution of the primary filter pack material (in the case of a filter-packed well). The screen openings must be small enough to retain most if not all of the formation or filter-pack materials, yet large enough to maintain ground-water flow velocities, from the well screen/filter pack interface back to the natural formation materials, of less than 0.10 ft/s (0.03 m/s). If well screen entrance velocities exceed 0.10 ft/s (0.03 m/s), turbulent flow conditions can occur, resulting in mobilization of sediment from the formation and reductions in well efficiency.

6.4.2 *Materials*—TThe well screen should be new, machine-slotted casing or continuous wrapped wire-wound screen composed of materials compatible with the monitoring environment, as determined by the site characterization program. The screen should be plugged at the bottom (unless a sediment sump is used), and the plug should generally be of the same material as the well screen. This assembly must have the capability to withstand well installation and development stresses without becoming dislodged or damaged. The length of the well screen open area should reflect the thickness of the target monitoring zone. Immediately prior to installation, the well screen should be cleaned (see either Practice D 5088 or Practice D 5608) with water from a source of known chemistry, if it is not certified clean by the manufacturer, and delivered, and maintained in a clean environment at the site.

NOTE 8—Well screens are most commonly composed of PVC or stainless steel. Stainless steel may be specified based on knowledge of the occurrence of microbially influenced corrosion in formations (specifically reducing or acid-producing conditions).

6.4.3 *Diameter*—TThe minimum nominal internal diameter of the well screen should be chosen based on factors specific to the particular application (such as the outside diameter of the purging and sampling device(s) to be used in the well). Well screens as small as 1/2-in. (1.27 cm) nominal diameter are available for use in monitoring well applications.

6.4.4 *Design*—The design of the well screen should be determined based on the grain size analysis (per Test Method D 422) of the interval to be monitored and the gradation of the primary filter pack material. In granular, non-cohesive formation materials that will fall in easily around the screen, filter packs can be developed from the native formation materials—filter pack materials foreign to the formation are not necessary. In these cases of naturally developed filter packs, the slot size of the well screen is determined using the grain size of the materials in the surrounding formation. The well screen slot size selected for this type of well completion should retain at least 70 % of formation materials—the finest 30 % of formation materials will be brought into the well during development, and the objectives of filter packing (to increase hydraulic conductivity immediately surrounding the well screen, and to promote easy flow of ground water into and through the screen) will be met. In wells in which a filter pack material of a selected grain size distribution is introduced from the surface, the screen slot size selected should retain at least 90 %, and preferably 99 %, of the primary filter pack materials. The method for determining the primary filter pack design is described in 6.3.3.

6.4.5 *Prepacked or Sleeved Well Screens*—An alternative to designing and installing filter pack and well screens separately is to use a pre-packed or sleeved screen assembly. A pre-packed well screen consists of an internal well screen, an external screen or filter medium support structure, and the filter medium contained between the screens, which together comprise an integrated structure. The internal and external screens are constructed of materials compatible with the monitored environment, and are usually of a common slot size specified by the well designer to retain the filter pack material. The filter pack is normally an inert (e.g., siliceous) granular material that has a grain-size distribution chosen to retain formation materials. A sleeved screen consists of a slotted pipe base over which a sleeve of stainless steel mesh filled with selected filter media is installed. Pre-packed or sleeved screens may be used for any formation conditions, but they are most often used where heaving, running or blowing sands make accurate placement of conventional well screens and filter packs difficult, or where predominantly fine-grained formation materials are encountered. In the latter case, using pre-packed or sleeved screens is the only practical means of ensuring that filter pack materials of the selected grain-size distribution (generally fine to very fine sands) are installed to completely surround the screen.

NOTE 9—The practice of using a single well screen/filter pack combination (e.g., 0.010 in. [0.254 mm]) well screen slot size with a 20/40 sand) for all wells, regardless of formation grain-size distribution, will result in siltation of the well and significant turbidity in samples when applied to formations finer than the recommended design. It will also result in the loss of filter pack, possible collapse of the screen, and invasion of overlying well construction materials (e.g., secondary filter pack, annular seal materials, grout) when applied to formations coarser than the recommended design. For these reasons, the universal application of a single well screen/filter pack combination to all formations is not recommended, and should be avoided.

6.5 *Riser*:

6.5.1 *Materials*—TThe riser should be new pipe composed of materials that will not alter the quality of water samples for the constituents of concern and that will stand up to long-term exposure to the monitoring environment, including potential contaminants. The riser should have adequate wall thickness and coupling strength to withstand the stresses imposed on it during well installation and development. Each section of riser should be cleaned (see either Practice D 5088 or Practice D 5608) using water from a source of known chemistry immediately prior to installation.

NOTE 10—Risers are generally constructed of PVC, galvanized steel or stainless steel.

6.5.2 *Diameter*—The minimum nominal internal diameter of the riser should be chosen based on the particular application. Risers as small as ½-in. (1.25-cm) in diameter are available for applications in monitoring wells.

6.5.3 *Joints (Couplings)*—Threaded joints are recommended. Glued or solvent-welded joints of any type are not recommended because glues and solvents may alter the chemistry of water samples. Because square profile flush joint

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threads (Specification F 480) are designed to be accompanied by O-ring seals at the joints, they do not require PTFE taping. However, tapered threaded joints should be PTFE taped to prevent leakage of water into the riser.

6.6 *Casing*—Where conditions warrant, the use of permanent casing installed to prevent communication between water-bearing zones is encouraged. The following subsections address both temporary and permanent casings.

6.6.1 *Materials*—The material type and minimum wall thickness of the casing should be adequate to withstand the forces of installation. All casing that is to remain as a permanent part of the installation (that is, in multi-cased wells) should be new and cleaned to be free of interior and exterior protective coatings.

NOTE 11—The exterior casing (temporary or permanent multi-cased) is generally composed of steel, although other appropriate materials may be used.

6.6.2 *Diameter*—Several different casing sizes may be required depending on the geologic formations penetrated. The diameter of the borehole and the well casing for conventionally filter packed wells should be selected so that a minimum annular space of 2 in. (5 cm) is maintained between the inside diameter of the casing and outside diameter of the riser to provide working space for a tremie pipe. For naturally developed wells and pre-packed or sleeved screen completions, this annular space requirement need not be met. In addition, the diameter of the casings in multi-cased wells should be selected so that a minimum annular space of 2 in. (5 cm) is maintained between the casing and the borehole (that is, a 2-in. [5 cm] diameter screen will require first setting a 6-in. [15.2 cm] diameter casing in a 10-in. [25.4 cm] diameter boring).

NOTE 12—Under difficult drilling conditions (collapsing soils, rock, or cobbles), it may be necessary to advance temporary casing. Under these conditions, a smaller annular space may be maintained.

6.6.3 *Joints (Couplings)*—The ends of each casing section should be either flush-threaded or beveled for welding.

6.7 *Sediment Sump*—A sediment sump, a length of blank pipe, generally of the same diameter and made of the same material as the riser and well screen -- may be affixed to the bottom of the screen, and capped with a bottom plug, to collect fine-grained material brought into the well by the process of well development. A drainage hole may be drilled in the bottom of the sump to prevent the sump from retaining water in the event that the water level outside the well falls below the bottom of the well screen. Because the sediment that collects in the sump may harbor geochemistry-altering microflora and reactive metal oxides, this sediment must be removed periodically to minimize the potential for sample chemical alteration.

6.8 *Protective Casing*:

6.8.1 *Materials*—Protective casings may be made of aluminum, mild steel, galvanized steel, stainless steel, cast iron, or structural plastic pipe. The protective casing should have a lid capable of being locked shut by a locking device or mechanism.

6.8.2 *Diameter*—The inside dimensions of the protective casing should be a minimum of 2 in. (5 cm) and preferably 4 in. (10 cm) larger than the nominal diameter of the riser to facilitate the installation and operation of sampling equipment.

6.9 *Annular Sealants*—TThe materials used to seal the annulus may be prepared as a slurry or used un-mixed in a dry pellet, granular, or chip form. Sealants should be selected to be compatible with ambient geologic, hydrogeologic, geochemical and climatic conditions and any man-induced conditions (e.g., subsurface contamination) anticipated during the life of the well.

6.9.1 *Bentonite*—Bentonite should be powdered, granular, pelletized, or chipped sodium montmorillonite from a commercial source, free of impurities that may adversely impact the water quality in the well. Pellets consist of roughly spherical units of moistened, compressed bentonite powder. Chips are large, irregularly shaped, and coarse granular units of bentonite free of additives. The diameter of pellets or chips selected for monitoring well construction should be less than one fifth the width of the annular space into which they are placed to reduce the potential for bridging. Granules consist of coarse to fine particles of unaltered bentonite, typically smaller than 0.2 in. (5.0 mm). It is recommended that the water chemistry of the formation in which the bentonite is intended for installation be evaluated to ensure that it is suitable to hydrate the bentonite. Some water-quality conditions (e.g., high chloride content, high concentrations of certain organic solvents or petroleum hydrocarbons) may inhibit the hydration of bentonite and result in an ineffective seal.

6.9.2 *Cement*—Each type of cement has slightly different characteristics that may be appropriate under various physical and chemical conditions. Cement should be one of the five Portland cement types that are specified in Specification C 150. The use of quick-setting cements containing additives is not recommended for use in monitoring well installation. Additives may leach from the cement and influence the chemistry of water samples collected from the monitoring well.

6.9.3 *Grout*—The grout backfill that is placed above the bentonite annular seal and secondary filters (see Fig. 1) is ordinarily a thick liquid slurry consisting of either a bentonite (powder or granules, or both) base and water, or a Portland cement base and water. Often, bentonite-based grouts are used when it is desired that the grout remain workable for extended periods of time during well construction or flexible (that is, to accommodate freeze-thaw cycles) during the life of the well. Cement-based grouts are often used when filling cracks in the surrounding geologic material, adherence to rock units, or a rigid setting is desired.

6.9.3.1 *Mixing*—The mixing (and placing) of a grout backfill should be performed with precisely recorded weights and volumes of materials, and according to procedures stipulated by the manufacturer that often include the order of component mixing. The grout should be thoroughly mixed with a paddle-type mechanical mixer or by recirculating the mix through a pump until all lumps are disintegrated. Lumpy grout should not be used in the construction of a monitoring well to prevent bridging within the tremie pipe.

NOTE 13—Lumps do not include lost circulation materials that may be added to the grout if excessive grout losses occur.

6.9.3.2 *Typical Bentonite-Based Grout*—When a bentonite-based grout is used, bentonite, usually unaltered, should be placed in the water through a venturi device. A typical

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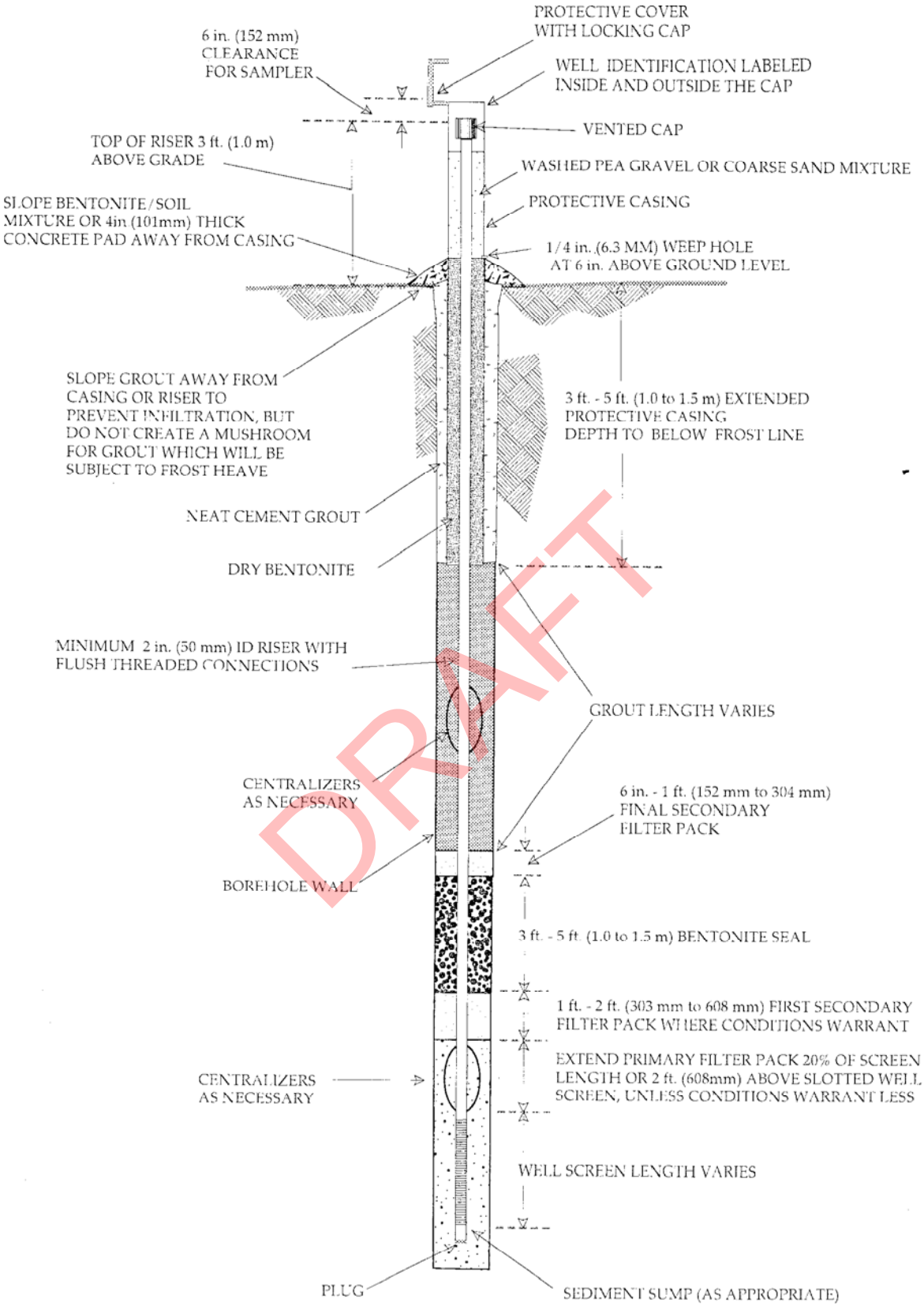

FIG. 1 Monitoring Well Design—Single–Cased Well

unbeneficiated bentonite-based grout consists of about 1 to 1.25 lb (0.57 kg) of unaltered bentonite to each 1 gal (3.8 L) of

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water. 100 % bentonite grouts should not be used for monitoring well annular sealants in the vadose zone of arid regions because of the possibility that they may desiccate. This could result in migration of water into the screened portion of the well from zones above the target monitoring zone.

NOTE 14—High solids bentonite grouts (minimum 20 % by weight with water) and other bentonite-based grouts may contain granular bentonite to increase the solids content and other components added under manufacturer's directions to either stiffen or retard stiffening of the mix. All additives to grouts should be evaluated for their effects on subsequent water samples.

6.9.3.3 *Typical Cement-Based Grout*—A typical cement-based grout consists of about 6 gal. (23 L) of water per 94-lb. (43-kg) bag of Type I Portland cement. Though not recommended because of the chemical incompatability of bentonite with cement (2, 3), from 3 to 8 % (by dry weight) of unaltered bentonite powder is often added after the initial mixing of cement and water to retard shrinkage and provide plasticity..

6.10 *Secondary Filter Packs*:

6.10.1 *Materials*—A secondary filter pack is a layer of material placed in the annulus between the primary filter pack and the bentonite seal, and/or between the bentonite seal and the grout backfill (see Fig. 1 and Fig. 2).

6.10.2 *Gradation*—The secondary filter pack should be uniformly graded fine sand with 100 % by weight passing the #30 U.S. Standard sieve, and less than 2 % by weight passing the #200 U.S. Standard sieve.

6.11 *Annular Seal and Filter Pack Installation Equipment*—The equipment used to install the annular seals and filter pack materials should be cleaned (if appropriate for the selected material) using water from a source of known quality prior to use. This procedure is performed to prevent the introduction of materials that may ultimately alter water quality samples.

7. Drilling Methods

7.1 The type of equipment required to create a stable, open, vertical borehole for installation of a monitoring well depends upon the site geology, hydrology, and the intended use of the data. Engineering and geological judgment and some knowledge of subsurface geological conditions at the site is required for the selection of the appropriate drilling method(s) utilized for drilling the exploratory soil borings and monitoring wells (see Guide D 6286). Appropriate drilling methods for investigating and installing monitoring wells at a site may include any one or a combination of several of the following methods: hollow-stem auger (Guide D 5784); direct (mud) rotary (Guide D 5783); direct air-rotary (Guide D 5782); direct rotary wireline casing advancement (Guide D 5876); dual-wall reverse-circulation rotary (Guide D 5781); cable-tool (Guide D 5875); or various casing advancement methods (Guide D 5872). Whenever feasible, it is advisable to utilize drilling procedures that do not require the introduction of water or drilling fluids into the borehole, and that optimize cuttings control at ground surface. Where the use of water or drilling fluid is unavoidable, the selected fluid should have as little impact as possible on the water samples for the constituents of interest. The chemistry of the fluid to be used should be evaluated to determine the potential for water quality sample alteration. In addition, care should be taken to remove as much drilling fluid as possible from the well and the surrounding formation during the well development process. It is recommended that if an air compressor is used, it should be equipped with an oil air filter or oil trap to minimize the potential for chemical alteration of ground-water samples collected after the well is installed. 8. Monitoring Well Installation

8. Monitoring Well Installation

8.1 *Stable Borehole*—A stable borehole must be constructed prior to attempting the installation of monitoring well screen and riser. Steps must be taken to stabilize the borehole before attempting installation if the borehole tends to cave or blow in, or both. Boreholes that are not straight or are partially obstructed should be corrected prior to attempting the installation procedures described herein.

8.2 *Assembly of Well Screen and Riser*:

8.2.1 *Handling*—TThe well screen, sediment sump, bottom plug and riser should be either certified clean from the manufacturer or steam-cleaned or high-pressure hot-water washed (whichever is appropriate for the selected material) using water from a source of known chemistry immediately prior to assembly. Personnel should take precautions to assure that grease, oil, or other contaminants that may ultimately alter the water sample do not contact any portion of the well screen and riser assembly. As one precaution, for example, personnel should wear a clean pair of cotton, nitrile or powder-free PVC (or equivalent) gloves while handling the assembly..

8.2.2 *Riser Joints (Couplings)*—Flush joint risers with square profile (Specification F 480) threads do not require PTFE taping to achieve a water tight seal; these joints should not be taped. O-rings made of a material of known chemistry, selected on the basis of compatibility with contaminants of concern and prevailing environmental conditions, should be used to assure a tight seal of flush-joint couplings. Couplings are often tightened by hand; however, if necessary, steam-cleaned or high-pressure water-cleaned wrenches may be utilized. Precautions should be taken to prevent damage to the threaded joints during installation, as such damage may promote leakage past the threads.

8.3 *Setting the Well Screen and Riser Assembly*—When the well screen and riser assembly is lowered to the predetermined level in the borehole and held in position, the assembly may require ballast to counteract the tendency to float in the borehole. Ballasting may be accomplished by filling the riser with water from a source of known and acceptable chemistry or, preferably, using water that was previously removed from the borehole. Alternatively, the riser may be slowly pushed into the fluid in the borehole with the aid of hydraulic rams on the drill rig and held in place as additional sections of riser are added to the column. Care must be taken to secure the riser assembly so that personnel safety is assured during the installation. The assembly must be installed straight and plumb, with centralizers installed at appropriate locations (typically every 20 to 30 ft [6 to 9 m]). Difficulty in maintaining a straight installation may be encountered where the weight of the well screen and riser assembly is significantly less than the buoyant force of the fluid in the borehole. The riser should extend above grade and be capped temporarily to deter entrance of foreign materials during final completion.

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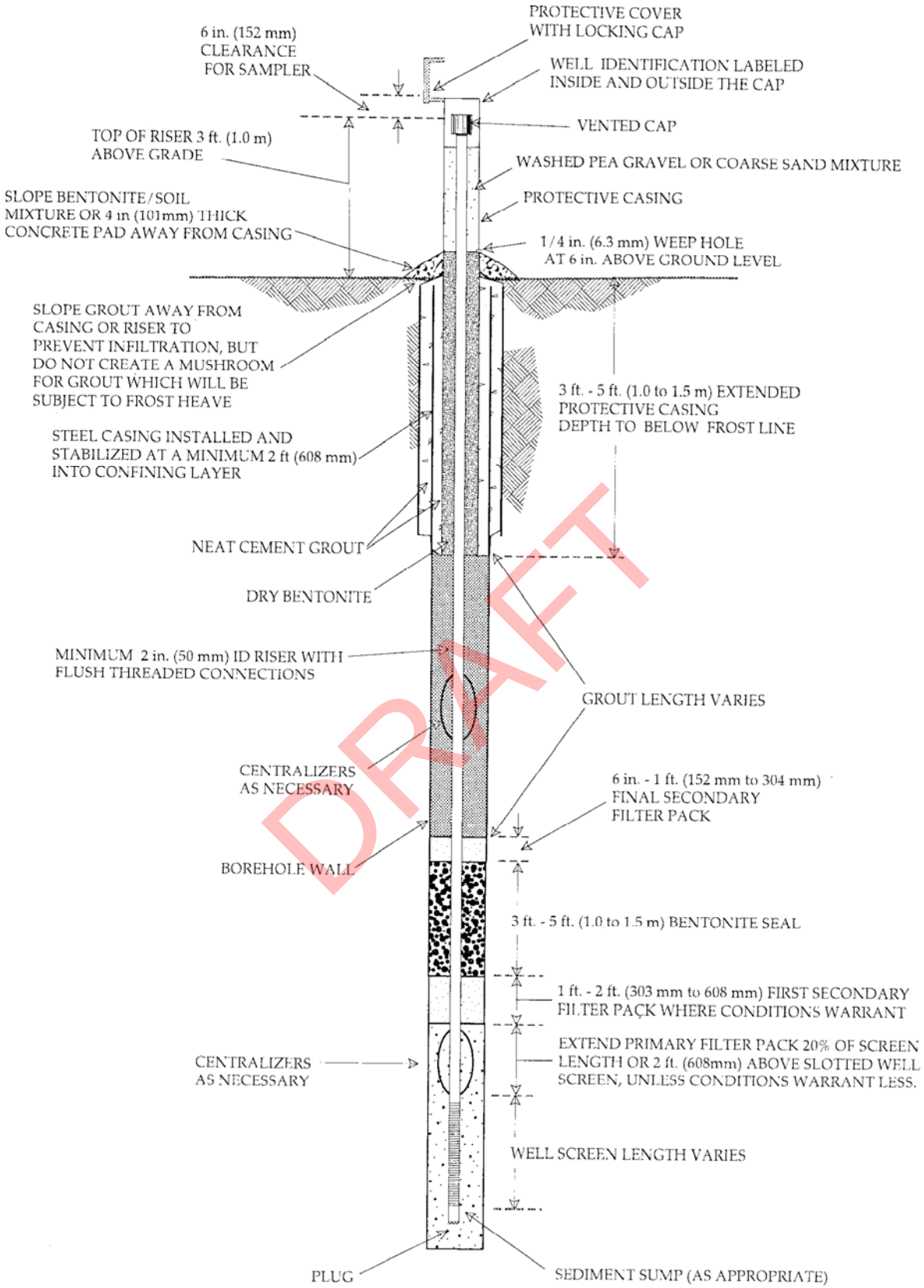

FIG. 2 Monitoring Well Design—Multi–Cased Well

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8.4 *Installation of the Primary Filter Pack*:

8.4.1 *Volume of Filter Pack*—TThe volume of filter pack required to fill the annular space between the well screen and borehole should be calculated, measured, and recorded on the well completion diagram during installation. To be effective, the filter pack should extend above the screen for a distance of about 20 % of the length of the well screen but not less than 2 ft. (0.6 m) (see Figs. 1 and 2). Where there is hydraulic connection between the zone to be monitored and the overlying strata, this upward extension should be gauged to prevent seepage from overlying hydrologic units into the filter pack. Seepage from other units may alter hydraulic head measurements or the chemistry of water samples collected from the well.

8.4.2 *Placement of Primary Filter Pack*—Placement of the well screen is preceded by placing no less than 2 % and no more than 10 % of the primary filter pack into the bottom of the borehole using a decontaminated, flush threaded, 1-in. (25-mm) minimum internal diameter tremie pipe. Alternatively, the filter pack may be added directly between the riser pipe and the auger or drive/temporary casing and the top of the filter pack located using a tamper or a weighted line. The well screen and riser assembly is then centered in the borehole. This can be done using one or more centralizer(s) or alternative centering devices located not more than 10 ft (3 m) above the bottom of the well screen (see Figs. 1 and 2). Centralizers should not be located in the well screen. The remaining primary filter pack is then placed in increments as the tremie is gradually raised or as the auger or drive/temporary casing is removed from the borehole. As primary filter pack material is poured into the tremie pipe, water from a source of known and acceptable chemistry may be added to help deliver the filter pack to the intended interval in the borehole. The tremie pipe or a weighed line can be used to measure the top of the primary filter pack as work progresses. If bridging of the primary filter pack material occurs, the bridged material should be broken mechanically prior to proceeding with the addition of more filter pack material. The elevation (or depth below ground surface), volume, and gradation of primary filter pack should be recorded on the well completion diagram (see Fig. 2 for an example).

8.4.3 *Withdrawal of the Temporary Casing/Augers*—If used, the drive/temporary casing or hollow stem auger is withdrawn, usually in stipulated increments. Care should be taken to avoid lifting the riser with the withdrawal of the temporary casing/augers. To limit borehole collapse in stable formations, the temporary casing or hollow stem auger is usually withdrawn until the lower-most point on the temporary casing or hollow stem auger is at least 2 ft (0.6 m), but no more than 5 ft (1.5 m) above the filter pack for unconsolidated materials; or at least 5 ft (1.5 m), but no more than 10 ft (3.0 m), for consolidated materials. In highly unstable formations, withdrawal intervals may be much less. After each increment, it should be ascertained that the primary filter pack has not been displaced during the withdrawal operation (using a weighed measuring device).

8.5 *Placement of First Secondary Filter*—A secondary filter pack may be installed above the primary filter pack to prevent the intrusion of the bentonite grout seal into the primary filter pack (see Figs. 1 and 2). To be effective, a measured and recorded volume of secondary filter material should be added to extend 1 to 2 ft (0.3 to 0.6 m) above the primary filter pack. As with the primary filter, a secondary filter must not extend into an overlying hydrologic unit (see 8.4.1). The well designer should evaluate the need for this filter pack by considering the gradation of the primary filter pack, the hydraulic heads between adjacent units, and the potential for grout intrusion into the primary filter pack. The secondary filter material is poured into the annular space through a decontaminated, flush threaded, 1-in. (25-mm) minimum internal diameter tremie pipe lowered to within 3 ft (1.0 m) of the placement interval. Water from a source of known and acceptable chemistry may be added to help deliver the filter pack to its intended location. The tremie pipe or a weighed line can be used to measure the top of the secondary filter pack as work progresses. The elevation (or depth below ground surface), volume, and gradation of the secondary filter pack should be recorded on the well completion diagram.

8.6 *Installation of the Bentonite Seal*—A bentonite pellet or a slurry seal is placed in the annulus between the borehole and the riser pipe on top of the secondary or primary filter pack (see Figs. 1 and 2). This seal retards the movement of cement-based grout backfill into the primary or secondary filter packs. To be effective, the bentonite seal should extend above the filter packs approximately 3 to 5 ft (1.0 to 1.5 m), depending on local conditions. The bentonite slurry seal should be installed using a positive displacement pump and a side-discharge tremie pipe lowered to the top of the filter pack. The tremie pipe should be raised slowly as the bentonite slurry fills the annular space. Bentonite pellets or chips may be poured from the surface and allowed to free-fall into the borehole. As a bentonite pellet or chip seal is poured into the borehole, a tamper may be necessary to tamp pellets or chips into place or to break bridges formed as the pellets or chips stick to the riser or the walls of the water-filled portion of the borehole. If the bentonite seal is installed above the water level in the borehole, granular bentonite should be used as the seal material – *bentonite pellets or chips should not be used in the unsaturated zone.* Granular bentonite should be poured into the borehole and installed in lifts of 2 in., then hydrated with water from a source of known chemistry. The tremie pipe or a weighed line can be used to measure the top of the bentonite seal as the work progresses. Sufficient time should be allowed for the bentonite pellet seal to hydrate or the slurry annular seal to expand prior to grouting the remaining annulus. The volume and elevation (or depth below ground surface) of the bentonite seal material should be measured and recorded on the well completion diagram.

8.7 *Final Secondary Filter Pack*—A 6-in. to 1-ft (0.15 to 0.3-m) secondary filter may be placed above the bentonite seal in the same manner described in 8.5 (see Figs. 1 and 2). This secondary filter pack will provide a layer over the bentonite seal to limit the downward movement of cement-based grout backfill into the bentonite seal. The volume, elevation (or depth

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below ground surface), and gradation of this final secondary filter pack should be documented on the well completion diagram.

8.8 *Grouting the Annular Space*:

8.8.1 *General*—Grouting procedures vary with the type of well design. The following procedures will apply to both single- and multi-cased monitoring wells. Paragraphs 8.8.2 and 8.8.3 detail those procedures unique to single- and multi-cased installations, respectively.

8.8.1.1 *Volume of Grout*—An ample volume of grout should be mixed on site to compensate for unexpected losses to the formation. The use of alternate grout materials, including grout containing gravel, may be necessary to control zones of high grout loss. The volume and location of grout used to backfill the remaining annular space is recorded on the well completion diagram.

8.8.1.2 *Grout Installation Procedures*—The grout should be pumped down hole through a side-discharge tremie pipe using a positive displacement pump (e.g., a diaphragm pump, moyno pump, or similar pump) to reduce the chance of leaving voids in the grout, and to displace any liquids and drill cuttings that may remain in the annulus. In very shallow wells, grouting may be accomplished by gravity feeding grout through a tremie pipe. With either method, grout should be introduced in one continuous operation until full-strength grout flows out of the borehole at the ground surface without evidence of drill cuttings, drilling fluid, or water.

8.8.1.3 *Grout Setting and Curing*—The riser should not be disturbed until the grout sets and cures for the amount of time necessary to prevent a break in the seal between the grout and riser. The amount of time required for the grout to set or cure will vary with the grout mix and ambient temperature and should be documented on the well completion diagram.

8.8.2 *Specific Procedures for Single-Cased Wells*—Grouting should begin at a level directly above the final secondary filter pack (see Fig. 1) if used, or above the bentonite pellet, chip or slurry seal. Grout should be pumped using a side-discharge tremie pipe to dissipate the fluid-pumping energy against the borehole wall and riser, reducing the potential for infiltration of grout into the primary filter pack. The tremie pipe should be kept full of grout from start to finish, with the discharge end of the pipe completely submerged as it is slowly and continuously lifted. Approximately 5 to 10 ft (1.5 to 3.0 m) of tremie pipe should remain submerged until grouting is complete. For deep installations or where the joints or couplings of the selected riser cannot withstand the collapse stress exerted by a full column of grout as it is installed, a staged grouting procedure may be used. If used, the drive/temporary casing or hollow-stem auger should be removed in increments immediately following each increment of grout installation and before the grout begins to set. If casing removal does not commence until grout pumping is completed, then, after the casing is removed, additional grout may be periodically pumped into the annular space to maintain a continuous column of grout up to the ground surface.

8.8.3 *Specific Procedures for Multi-Cased Wells*—If the outer casing of a multi-cased well cannot be driven to form a tight seal between the surrounding stratum (strata) and the casing, it should be installed in a pre-drilled borehole. After the borehole has penetrated not less than 2 ft. (0.6 m) of the first targeted confining stratum, the outer casing should be lowered to the bottom of the boring and the annular space pressure grouted. Pressure grouting requires the use of a grout shoe or packer installed at the end of the outer casing to prevent grout from moving up into the casing. The grout must be allowed to cure and form a seal between the casing and the borehole prior to advancing the hole to the next hydrologic unit. This procedure is repeated as necessary to advance the borehole to the desired depth. Upon reaching the final depth, the riser and screen should be set through the inner casing. After placement of the filter packs and bentonite seal, the remaining annular space is grouted as described in 8.8.2 (see Fig. 2).

NOTE 15—When using a packer, pressure may build up during grout injection and force grout up the sides of the packer and into the casing.

8.9 *Well Protection*—Well protection refers specifically to installations made at the ground surface to deter unauthorized entry to the monitoring well, to prevent damage to or destruction of the well, and to prevent surface water from entering the annulus. The methods described in Practice D 5787 should be used for well protection.

8.9.1 *Protective Casing*—Protective casing should be used for all monitoring well installations. In areas that experience frost heaving, the protective casing should extend from below the depth of frost penetration (3 to 5 ft [1.0 to 1.5 m] below grade, depending on local conditions), to slightly above the top of the well casing. The protective casing should be initially placed before final set of the grout. The protective casing should be sealed and immobilized in concrete placed around the outside of the protective casing above the set grout. The protective casing should be stabilized in a position concentric with the riser (see Figs. 3 and 1). Sufficient clearance, usually 6 in. (0.15 m) should be maintained between the lid of the protective casing and the top of the riser to accommodate sampling equipment. A ¼-in. (6.3-mm) ddiameter weep hole should be drilled in the protective casing approximately 6 in. (15 cm) above ground surface to permit water to drain out of the annular space between the protective casing and the riser. In cold climates, this hole will also prevent water freezing between the protective casing and the well casing. Dry bentonite pellets, granules, or chips should then be placed in the

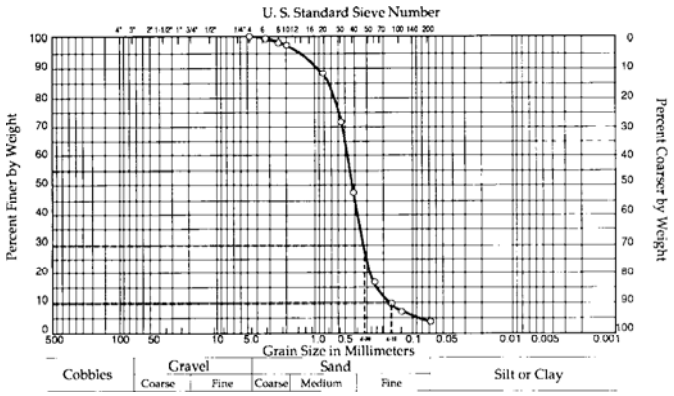

FIG. 3 Example Grading Curve for Design of Monitoring Well Screens

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annular space below ground level within the protective casing. Coarse sand or pea gravel or both should be placed in the annular space above the dry bentonite pellets and to just above the weep hole to prevent entry of insects. All materials chosen should be documented on the well completion diagram. The monitoring well identification number should be clearly visible on the inside and outside of the protective casing.

8.9.2 *Completion of Surface Installation*—The well protection installation may be completed in one of three ways:

8.9.2.1 In areas subject to frost heave, place a soil or bentonite/sand layer adjacent to the protective casing sloped to direct water drainage away from the well.

8.9.2.2 In regions not subject to frost heave, a concrete pad, sloped slightly to provide water drainage away from the well, should be placed around the installation.

8.9.2.3 Where monitoring well protection must be installed flush with the ground, an internal cap should be fitted on top of the riser within the manhole or vault. This cap should be leak-proof so that if the vault or manhole should fill with water, the water will not enter the well casing. Ideally, the manhole cover cap should also be leak-proof..

8.9.3 *Additional Protection*—In areas where there is a high probability of damaging the well (high traffic, heavy equipment, poor visibility), it may be necessary to enhance the normal protection of the monitoring well through the use of posts, markers, signs, or other means, as described in Practice D 5787. The level of protection should meet the damage threat posed by the location of the well.

9. Well Development

9.1 *General*—Well development serves to remove fine-grained material from the well screen and filter pack that may otherwise interfere with water quality analyses, to restore the formation properties disturbed during the drilling process, and to improve the hydraulic characteristics of the filter pack and hydraulic communication between the well and the hydrologic unit adjacent to the well screen. Methods of well development vary with the physical characteristics of hydrologic units in which the monitoring well is screened and with the drilling method used.

9.2 *Development Methods and Procedures*—The methods and procedures for well development described in Guide D 5521 should be followed to ensure a proper well completion.

9.3 *Timing and Duration of Well Development*—Well development should begin either after the riser, well screen and filter pack are installed and before the bentonite seal and grout are installed (the preferred time), or after the monitoring well is completely installed and the grout has cured or set. In the former case, the installer may add filter pack material to the borehole before the bentonite seal is installed to compensate for settlement that typically occurs during the development process. This allows the installer to maintain the desired separation between the top of the screen and the bentonite seal. In the latter case, the possibility exists that settlement of the filter pack may result in the bentonite seal settling into the top of the screen. Development should be continued until representative water, free of the drilling fluids, cuttings, or other materials introduced or produced during well construction, is obtained. Representative water is assumed to have been obtained when turbidity readings stabilize and the water is visually clear of suspended solids. The minimum duration of well development will vary with the method used to develop the well. The timing and duration of well development and the turbidity measurements should be recorded on the well completion diagram.

9.4 *Well Recovery Test*—A well recovery test should be performed immediately after and in conjunction with well development. The well recovery test provides an indication of well performance and provides data for estimating the hydraulic conductivity of the screened hydrologic unit. Readings should be taken at intervals suggested in Table 2 until the well has recovered to 90 % of its static water level.

NOTE 16—If a monitoring well does not recover sufficiently for sampling within a 24-hr period and the well has been properly developed, the installation should not generally be used as a monitoring well for detecting or assessing low level organic constituents or trace metals. The installation may, however, be used for long-term water-level monitoring if measurements of short-frequency water-level changes are not required.

10. Installation Survey

10.1 *General*—The vertical and horizontal position of each monitoring well in the monitoring system should be surveyed and subsequently mapped by a licensed surveyor. The well location map should include the location of all monitoring wells in the system and their respective identification numbers, elevations of the top of riser position to be used as the reference point for water-level measurements, and the elevations of the ground surface protective installations. The locations and elevations of all permanent benchmark(s) and pertinent boundary marker(s) located on-site or used in the survey should also be noted on the map.

10.2 *Water-Level Measurement Reference*—The water-level measurement reference point should be permanently marked, for example, by cutting a V-notch into the top edge of the riser pipe. This reference point should be surveyed in reference to the nearest NAVD reference point.

10.3 *Location Coordinates*—The horizontal location of all monitoring wells (active or decommissioned) should be surveyed by reference to a standardized survey grid or by metes and bounds.

10.4 *Borehole Deviation Survey*—A borehole deviation survey, to determine the direction and distance of the bottom of the well relative to the top of the well and points in between, should be completed in wells deeper than 100 feet and in wells installed in dipping formations.

11. Monitoring Well Network Report

11.1 TTo demonstrate that the goals set forth in the Scope have been met, a monitoring well network report should be prepared. This report should:

TABLE 2 Suggested Recording Intervals for Well Recovery Tests

Time Since Starting Test	Time Interval
0 to 15 min	1 min
15 to 50 min	5 min
50 to 100 min	10 min
100 to 300 min (5 h)	30 min
300 to 1440 min (24 h)	60 min

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11.1.1 Locate the area investigated in terms pertinent to the project. This should include sketch maps or aerial photos on which the exploratory borings, piezometers, sample areas, and monitoring wells are located, as well as topographic items relevant to the determination of the various soil and rock types, such as contours, streambeds, etc. Where feasible, include a geologic map and geologic cross sections of the area being investigated.

11.1.2 Include copies of all well boring test pits and exploratory borehole logs, initial and post-completion water levels, all laboratory test results, and all well completion diagrams.

11.1.3 Include the well installation survey.

11.1.4 Describe and relate the findings obtained in the initial reconnaissance and field investigation (Section 5) to the design and installation procedures selected (Sections 7-9) and the surveyed locations (Section 10).

11.1.5 This report should include a recommended decommissioning procedure that is consistent with those described in Guide D 5299 and/or with applicable regulatory requirements.

12. Keywords

12.1 aquifer; borehole drilling; geophysical exploration; ground water; monitoring well; site investigation

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CES-SOP-03: Equipment Decontamination

All field equipment will be decontaminated per the following Alaska Department of Environmental Conservation (ADEC) guidance documents:

- **Field Sampling Guidance (ADEC, 2017)**
- **Underground Storage Tanks Procedures Manual: Guidance for Treatment of Petroleum-Contaminated Soil and Groundwater and Standard Sampling Procedures (ADEC, 2017)**

Depending on the contaminant, wash water and rinsate solutions may need to be collected in appropriate containers and disposed of properly in accordance with federal, state, and local regulations. Proposed decontamination water management needs to be described in work plans.

Decontaminate all reusable equipment such as steel tapes, well sounders, transducers, and water quality probes after each sampling point using a stiff brush and a solution of water and laboratory-grade detergent. An appropriate solvent may be used to remove heavy contaminant residues from the sampling tools. If necessary, sampling equipment can be sterilized in the field with chemical disinfectants, (e.g., detergents, hydrogen peroxide, sodium hypochlorite, ethanol, etc.) or heat (flame) sterilization. Rinse tools twice in clean water and again with distilled or deionized water.

Properly collect, store, and dispose of solvent waste and wash water in accordance with hazardous waste regulations, if applicable, and the CSP site-specific approved work plan. Clean drill auger sections, split spoons, and drive hammers that come in contact with bore holes before use and between borings. Scrub tools with a stiff brush in a solution of water and laboratory-grade detergent. High pressure water or steam may also be used.

Visibly contaminated decontamination water for sites with petroleum hydrocarbons may be containerized for off-site shipment, or with CSP site-specific approval, filtered on-site and reapplied directly to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies. If not visibly contaminated, decontamination water may be re-applied directly to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies, if approved in a CSP site-specific work plan.

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Non-dedicated Equipment Decontamination Procedures

1. Remove as much gross contamination (such as pieces of soil) as possible off equipment at the sampling site.
2. Wash water-resistant equipment thoroughly and vigorously with potable water containing non-phosphate laboratory-grade detergent such as Alconox, or equivalent, and using a bristle brush or similar utensil to remove any remaining residual contamination.
3. Rinse equipment thoroughly with potable water (1st rinse).
4. Rinse equipment thoroughly with distilled or deionized water (2nd rinse).
5. For sensitive field instruments, rinse equipment with distilled, deionized, or American Society for Testing and Materials (ASTM) reagent grade water (3rd rinse).
6. Air dry at a location where dust or other fugitive contaminants may not contact the sample equipment. Alternatively, wet equipment maybe dried with a clean, disposable paper towel to assist the drying process. All equipment should be dry before reuse.
7. Store equipment in new, unused plastic bags to protect the decontaminated equipment from fugitive contaminates before reuse.

Drilling and Subsurface Soil Sampling Equipment Decontamination Procedures

Drilling equipment and associated materials will be decontaminated by the drilling contractor prior to any drilling operations and between borings. Decontaminate tools used for soil sampling (for example, split spoon samplers) before and between collecting any analytical samples.

1. Remove as much gross contamination as possible off equipment at the sampling site.
2. Wash equipment thoroughly and vigorously with high-temperature potable water using a high-pressure washer and/or steam cleaner. A bristle brush is also suggested to remove any persistent gross contamination.
3. Rinse equipment twice thoroughly with potable water (1st and 2nd rinse).
4. Air dry at a location where dust or other fugitive contaminants may not contact the sample equipment. All equipment should be dry before reuse.
5. Store decontaminated equipment at a location away from any potential exposure from fugitive contamination.

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CES-SOP-04: Instrument Calibration, Well Purging and Field Water Quality Measurements

All instrument calibration and well sampling and field water qaulity measurements will be performed per the following Alaska Department of Environmental Conservation (ADEC) guidance documents:

- **Field Sampling Guidance (ADEC, 2017)**
- **Underground Storage Tanks Procedures Manual: Guidance for Treatment of Petroleum-Contaminated Soil and Groundwater and Standard Sampling Procedures (ADEC, 2017)**

Instrument Calibration

All field instruments must be calibrated prior to each project according to manufacturer's specifications and instrument calibration must be checked and documented on-site on a daily basis. Certain field screening parameters may require more frequent calibrations depending on site conditions. CES will retain a reference copy of manufacturer's operating instructions in the field. All instrument users must be trained in routine maintenance and operation. Calibration standard(s), dates, times and all calibration results must be recorded in the field record or log book.

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YSI 556 MPS

Multi Probe System

Operations Manual

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1. Safety

1.1 General Information

Read all safety information in this manual carefully before using the YSI 556 Multi-Probe System (MPS). Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review Appendix D Health and Safety.

⚠ **WARNING**

Warnings are used in this manual when misuse of the instrument could result in death or serious injury to a person.

⚠ **CAUTION**

Cautions are used in this manual when misuse of the instrument could result in mild or serious injury to a person and/or damage to equipment.

⚠ **IMPORTANT SAFETY INSTRUCTIONS!**

⚠ **SAVE THESE INSTRUCTIONS!**

⚠ In essence, the most important safety rule for use of the YSI 556 MPS is to utilize the instrument ONLY for purposes documented in this manual. This is particularly true of the YSI 6117 rechargeable battery pack that contains nickel metal hydride (NiMH) batteries. The user should be certain to read all of the safety precautions outlined below before using the instrument.

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⚠ Batteries

⚠ This instrument is powered by alkaline or optional nickel-metal hydride batteries, which the user must remove and dispose of when the batteries no longer power the instrument. Disposal requirements vary by country and region, and users are expected to understand and follow the battery disposal requirements for their specific locale.

The circuit board in this instrument contains a manganese dioxide lithium "coin cell" battery that must be in place for continuity of power to memory devices on the board. This battery is not user serviceable or replaceable.

When appropriate, an authorized YSI service center will remove this battery and properly dispose of it, per service and repair policies.

⚠YSI Rechargeable Battery Pack Safety Information

⚠Restrictions on Usage

1. Never dispose of the battery pack in a fire.
2. Do not attempt to disassemble the YSI 6117 battery pack
3. Do not tamper with any of the electronic components or the batteries within the battery pack. Tampering with either the electronic circuitry or the batteries will result in the voiding of the warranty and the compromising of the system performance, but, more importantly, can cause safety hazards which result from overcharging such as overheating, venting of gas, and loss of corrosive electrolyte.
4. Do not charge the battery pack outside the 0–40°C temperature range.
5. Do not use or store the battery at high temperature, such as in strong direct sunlight, in cars during hot weather, or directly in front of heaters.
6. Do not expose the battery pack to water or allow the terminals to become damp.
7. Avoid striking or dropping the battery pack. If the pack appears to have sustained damage from these actions or malfunctions after an impact or drop, the user should not attempt to repair the unit. Instead, contact YSI Customer Service. Refer to *Appendix E Customer Service.*
8. If the battery pack is removed from the YSI 556 MPS, do not store it in pockets or packaging where metallic objects such as keys can short between the positive and negative terminals.

⚠Precautions for Users with Small Children.

Keep the battery pack out of reach of babies and small children.

⚠Danger Notifications – Misuse creates a STRONG possibility of death or serious injury.

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FAILURE TO CAREFULLY OBSERVE THE FOLLOWING PROCEDURES AND PRECAUTIONS CAN RESULT IN LEAKAGE OF BATTERY FLUID, HEAT GENERATION, BURSTING, AND SERIOUS PERSONAL INJURY.

1. Never dispose of the battery pack in a fire or in heat.
2. Never allow the positive and negative terminals of the battery pack to become shorted or connected with electrically conductive materials. When the battery pack has been removed from the YSI 556 MPS, store it in a heavy plastic bag to prevent accidental shorting of the terminals.
3. Never disassemble the battery pack and do not tamper with any of the electronic components or the batteries within the battery pack. The battery pack is equipped with a variety of safety features. Accidental deactivation of any of these safety features can cause a serious hazard to the user.
4. The NiMH batteries in the battery pack contain a strong alkaline solution (electrolyte). The alkaline solution is extremely corrosive and will cause damage to skin or other tissues. If any fluid from the battery pack comes in contact with a user's eyes, immediately flush with clean water and consult a physician immediately. The alkaline solution can damage eyes and lead to permanent loss of eyesight.

⚠ Warning Notifications – Misuse creates a possibility of death or serious injury

1. Do not allow the battery pack to contact freshwater, seawater, or other oxidizing reagents that might cause rust and result in heat generation. If a battery becomes rusted, the gas release vent may no longer operate and this failure can result in bursting.
2. If electrolyte from the battery pack contacts the skin or clothing, thoroughly wash the area immediately with clean water. The battery fluid can irritate the skin.

⚠ Caution Notifications – Misuse creates a possibility of mild or serious injury or damage to the equipment.

1. Do not strike or drop the battery pack. If any impact damage to the battery pack is suspected, contact YSI Customer Service. Refer to *Appendix E Customer Service*.

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2. Store the battery pack out of reach of babies and small children.
3. Store the battery pack between the temperatures of -20 and 30°C.
4. Before using the battery pack, be sure to read the operation manual and all precautions carefully. Then store this information carefully to use as a reference when the need arises.

⚠YSI 616 Cigarette Lighter Charger Safety Information

1. This section contains important safety and operating instructions for the YSI 556 MPS cigarette lighter battery charger (YSI 616; RadioShack Number 270-1533E). BE SURE TO SAVE THESE INSTRUCTIONS.
2. Before using the YSI 616 cigarette lighter charger, read all instructions and cautionary markings on battery charger, battery pack, and YSI 556 MPS.
3. Charge the YSI 6117 battery pack with the YSI 616 cigarette lighter charger ONLY when the YSI 6117 is installed in the YSI 556 MPS.
4. Do not expose charger to rain, moisture, or snow.
5. Use of an attachment not recommended or sold by the battery charger manufacturer may result in a risk of fire, electric shock, or injury to persons.
6. To reduce risk of damage to cigarette lighter and cord, pull by cigarette lighter rather than cord when disconnecting charger.
7. Make sure that the cord is located so that it will not be stepped on, tripped over, or otherwise subjected to damage or stress.
8. Do not operate charger with damaged cord or cigarette lighter connector – replace it immediately.
9. Do not operate charger if it has received a sharp blow, been dropped, or otherwise damaged in any way; contact YSI Customer Service. Refer to *Appendix E Customer Service*.
10. Do not disassemble charger other than to change the fuse as instructed. Replace the part or send it to YSI Product Service if repair is required (refer to *Appendix E Customer Service*). Incorrect reassembly may result in a risk of electric shock or fire.

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11. To reduce risk of electric shock, unplug charger before attempting any maintenance or cleaning. Turning off controls will not reduce this risk.

⚠ YSI 556 MPS Water Leakage Safety Information

The YSI 556 MPS has been tested and shown to comply with IP67 criterion, i.e. submersion in 1 meter of water for 30 minutes with no leakage into either the battery compartment or the main case. However, if the instrument is submersed for periods of time in excess of 30 minutes, leakage may occur with subsequent damage to the batteries, the rechargeable battery pack circuitry, and/or the electronics in the main case.

If leakage into the battery compartment is observed when using alkaline C cells, remove batteries, dispose of batteries properly, and dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

If leakage into the battery compartment is observed when using the YSI rechargeable battery pack, remove the battery assembly and set aside to dry. Return the battery pack to YSI Product Service for evaluation of possible damage. Finally dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** If water has contacted the rechargeable battery pack, do not attempt to reuse it until it has been evaluated by YSI Product Service (refer to Appendix E Customer Service). Failure to follow this precaution can result in serious injury to the user.

If it is suspected that leakage into the main cavity of the case has occurred, remove the batteries immediately and return the instrument to YSI Product Service for damage assessment. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** Under no circumstances should the user attempt to open the main case.

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2. General Information

2.1 Description

The rugged and reliable YSI 556 MPS (Multi-Probe System) combines the versatility of an easy-to-use, easy-to-read handheld unit with all the functionality of a multi-parameter system. Featuring a waterproof, impact-resistant case, the YSI 556 MPS simultaneously measures dissolved oxygen, conductivity, temperature, and optional pH and ORP. A simple cellular phone style keypad and large display make the instrument easy to use. The YSI 556 MPS is compatible with YSI EcoWatch™ for Windows™ software.

The YSI 556 MPS assists the user in conforming to Good Laboratory Practice (GLP) standards which help ensure that quality control/quality assurance methods are followed. Battery life is displayed with a fuel gauge, and the user can choose standard alkaline batteries or an optional rechargeable battery pack.

The 1.5 MB memory can store more than 49,000 data sets. Other options include a flow cell and barometer. The internal barometer can be user-calibrated and displayed along with other data, used in dissolved oxygen calibrations, and logged to memory for tracking changes in barometric pressure.

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Features

- Waterproof -meets IP67 specifications
- Field-replaceable DO electrode module; pH and pH/ORP sensors
- Compatible with EcoWatch™ for Windows™ data analysis software
- Assists with Good Laboratory Practice Standards (GLP)
- Choice of DO membrane material for different applications
- Easy-to-use, screw-on cap DO membranes
- User-upgradeable software from YSI website
- Three-year warranty on the instrument; one-year on the probe modules
- Available with 4,10, and 20 m cable lengths
- Stores over 49,000 data sets, time and date stamped

- Auto temperature compensating display contrast
- Optional barometer
- Optional rechargeable battery pack or standard alkaline batteries

2.2 Unpacking the Instrument

1. Remove the instrument from the shipping box. Note that the probe module and sensors are shipped in a separate box and will be unpacked later in Section *3.2 Unpacking the Probe Module*

   NOTE: Do not discard any parts of supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

   NOTE: If any parts are missing or damaged, contact your YSI Service Center immediately. Refer to Appendix E Customer Service or www.ysi.com.

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2.3 Features of the YSI 556 Multi-Probe System

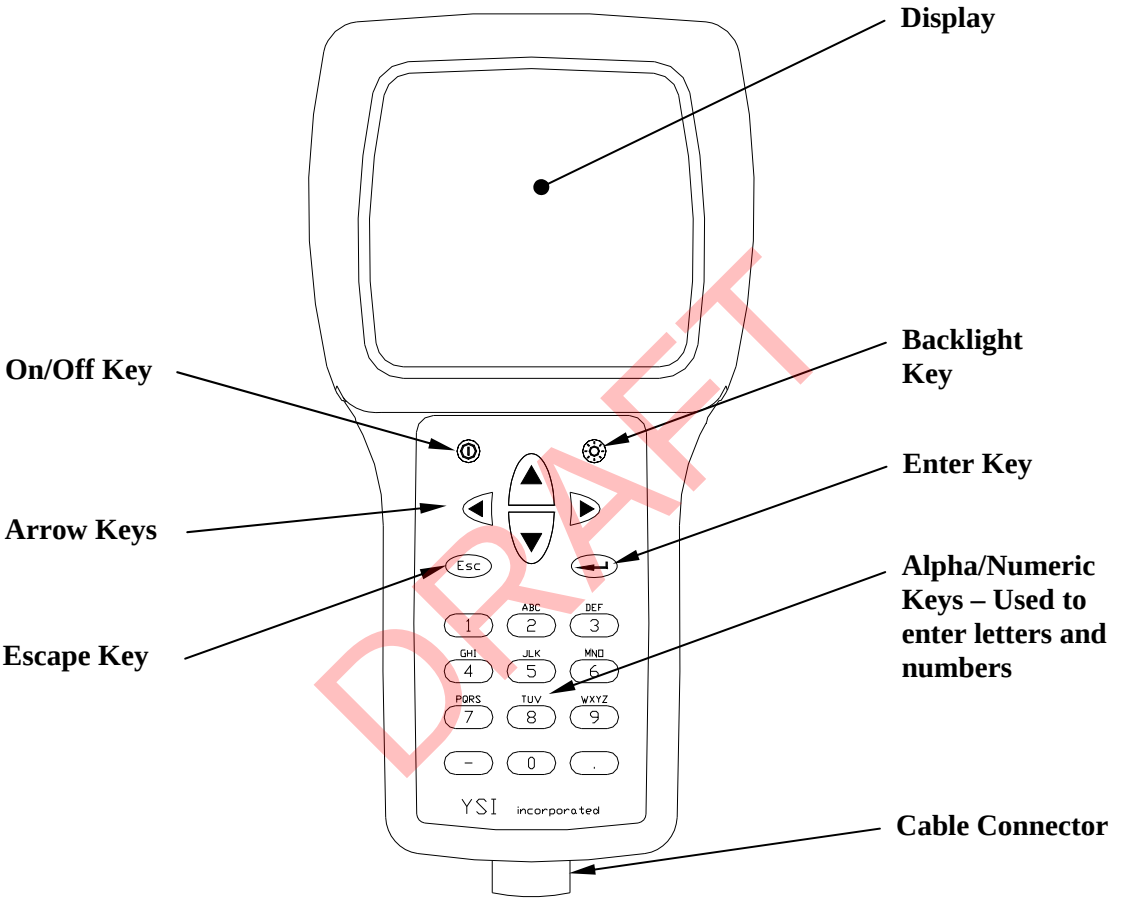

Figure 2.1 Front View of YSI 556 MPS

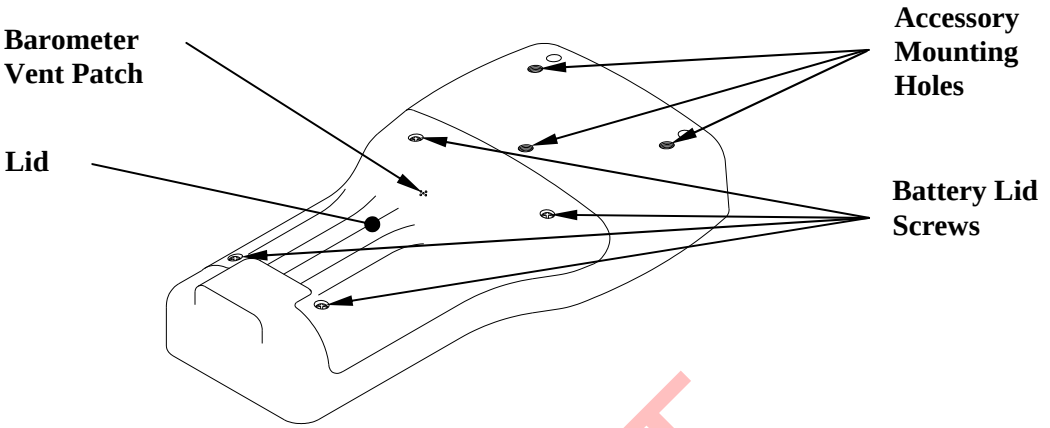

Figure 2.2 Back View of YSI 556 MPS

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2.4 Batteries

2.4.1 Battery Life

Standard Alkaline Batteries

With the standard battery configuration of 4 alkaline C cells, the YSI 556 MPS will operate continuously for approximately 180 hours. Assuming a standard usage pattern when sampling of 3 hours of “on time” in a typical day, the alkaline cells will last approximately 60 days.

Optional Rechargeable Battery Pack

When fully charged, the optional rechargeable battery pack will provide approximately 50 hours of battery life.

2.4.2 Inserting 4 C Batteries

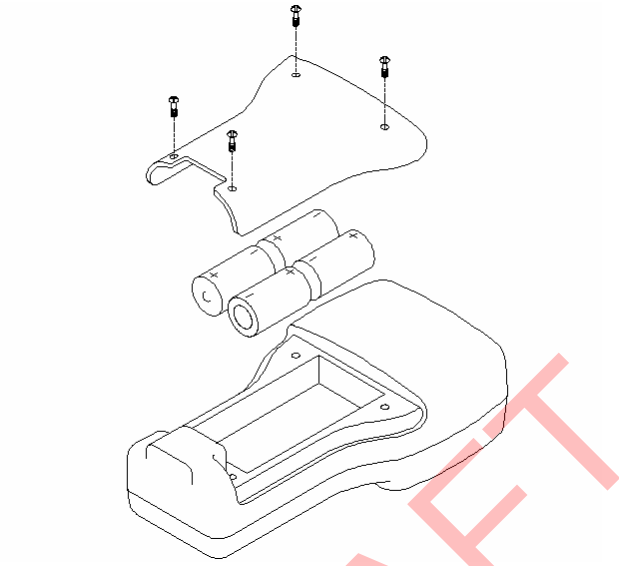

Figure 2.1 Inserting C Cells

⚠ **CAUTION: Install batteries properly to avoid damage to the instrument**.

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.
2. Remove the battery lid.
3. Insert four C batteries between the clips following the polarity (+ and -) labels on the bottom of the battery compartment.
4. Check gasket for proper placement on the battery lid.
5. Replace the battery lid and tighten the 4 screws securely and evenly.

NOTE: Do not over-tighten the screws.

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2.4.3 Inserting Optional Rechargeable Battery Pack

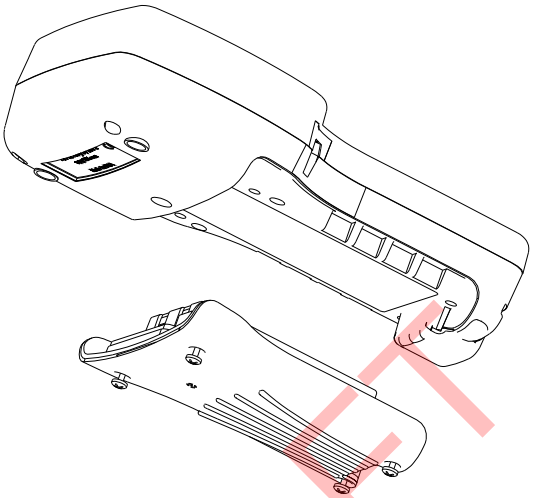

Figure 2.2 Inserting Battery Pack

⚠ **CAUTION: Read all cautions and warning that come with the battery pack before using the battery pack.**

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.
2. Remove the C battery lid and store for future use. Remove C batteries, if installed.
3. Install the rechargeable battery pack and lid and tighten the 4 screws securely and evenly.

NOTE: Do not over tighten the screws.

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2.4.4 Charging the Optional Rechargeable Battery Pack

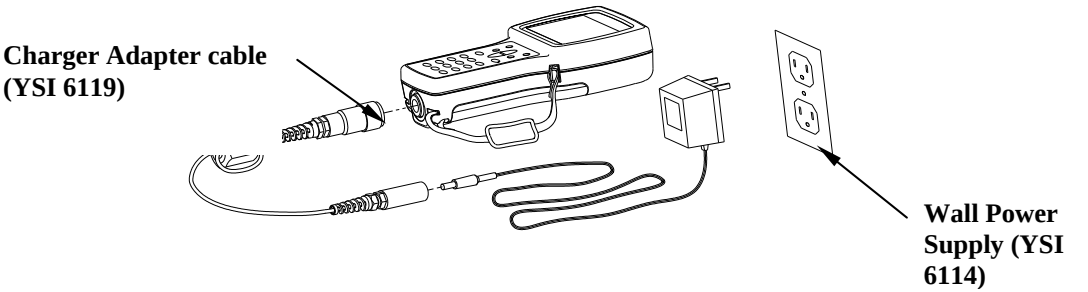

Figure 2.3 Charging the Battery Pack

⚠ **CAUTION: Do not use or store the battery pack at extreme temperatures such as in strong direct sunlight, in cars during hot weather or close to heaters.**

1. Install the rechargeable battery pack into the instrument as described in Section *2.4.3 Inserting Optional Rechargeable Battery Pack*.

2. Attach the charger adapter cable (YSI 6119) to the instrument.

NOTE: Wall power supplies for use in countries outside the US and Canada can be found in *Appendix B Instrument Accessories.*

3. Insert the barrel connector of the wall power supply into the barrel of the adapter cable.

⚠ **CAUTION: Do not charge the battery pack continuously for more than 48 hours.**

⚠ **CAUTION: Do not drop or expose to water.**

⚠ **CAUTION: Do no charge the battery pack at temperatures below 0°C or above 40°C.**

4. Plug the wall power supply into an AC power outlet for approximately 2 hours to obtain an 80% to 90% charge for 6 hours to get a full charge.

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NOTE: The battery pack can be recharged whether the instrument is on or off.

2.4.5 Storing the Battery Pack

Remove the battery pack from the instrument when the instrument will not be used for extended periods of time to prevent over discharge of the battery pack.

Store the battery pack in a heavy plastic bag to prevent accidental shorting of the terminals. Store between –20 and 30°C.

2.4.6 Optional Cigarette Lighter Charger

⚠ **CAUTION: Read all warnings and cautions that come with the charger before using the charger.**

⚠ **CAUTION: Only use cigarette lighter charger when rechargeable battery pack is inserted into instrument.**

⚠ **CAUTION: Do not mishandle cigarette lighter charger. Do not expose to moisture.**

1. Plug the barrel connector of the cigarette lighter charger into the mating end of the YSI 6119 Charger Adapter Cable.

2. Attach the MS-19 end of the YSI 6119 Charger Adapter Cable to the instrument.

3. Make one of the following modifications to the other end of the charger:

Slide the adapter ring off the plug to use the device with an American or Japanese vehicle.

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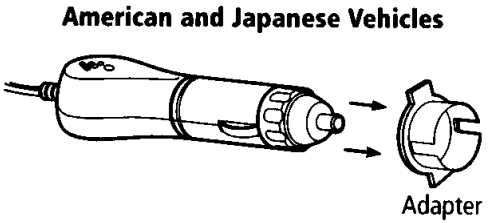

Figure 2.1 Charger Plug Adapter Use

Leave the adapter ring on the plug and position it so that the slots on the adapter ring line up with the plug's spring clips to use the device on a European vehicle.

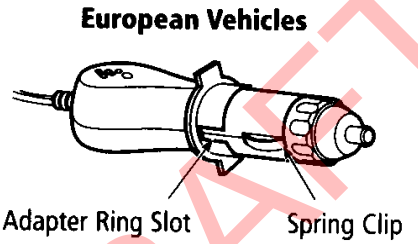

Figure 2.2 European Charger Plug Adapter Use

NOTE: If the charger stops working properly, refer to Section 13 *Troubleshooting.*

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2.5 Power On

Press and release the on/off button in the upper left corner of the instrument keypad to turn the instrument on or off. See Figure2.1 Front View of YSI 556 MPS.

2.6 Setting Display Contrast

The display contrast automatically compensates for temperature changes. However, under extreme temperature conditions you may wish to optimize the display by manual adjustment as follows:

1. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “up” arrow to increase (darken) the contrast.

2. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “down” arrow to decrease (lighten) the contrast.

2.7 Backlight

Press and *release* the backlight key in the upper right corner of the keypad to turn the backlight on or off. See Figure 2.1 Front View of YSI 556 MPS.

NOTE: The backlight turns off automatically after two minutes of non-use.

2.8 General Screen Features

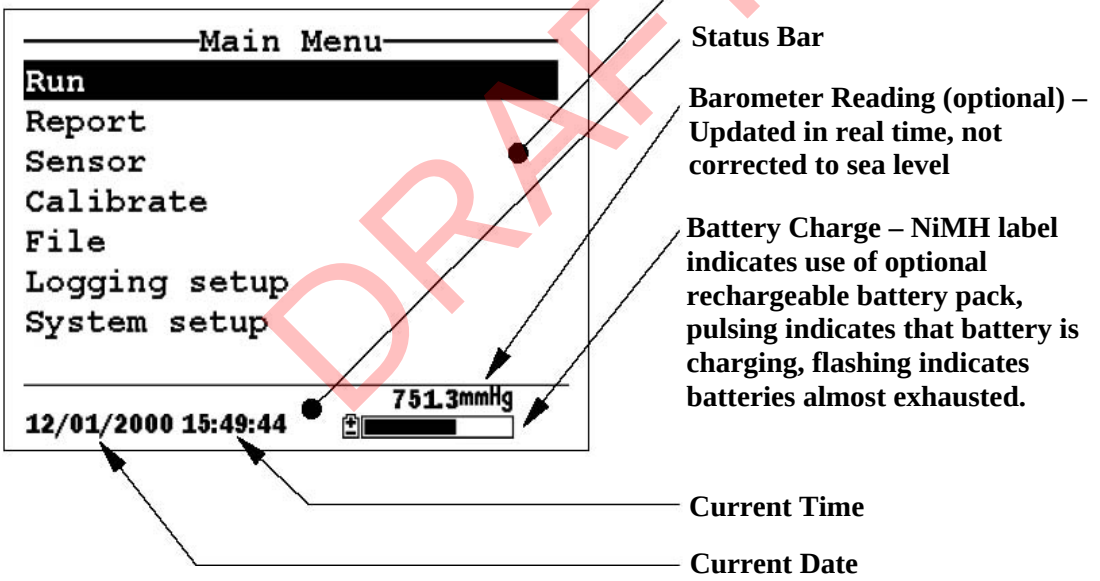

Figure 2.4 Main Screen Menu

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2.9 Keypad Use

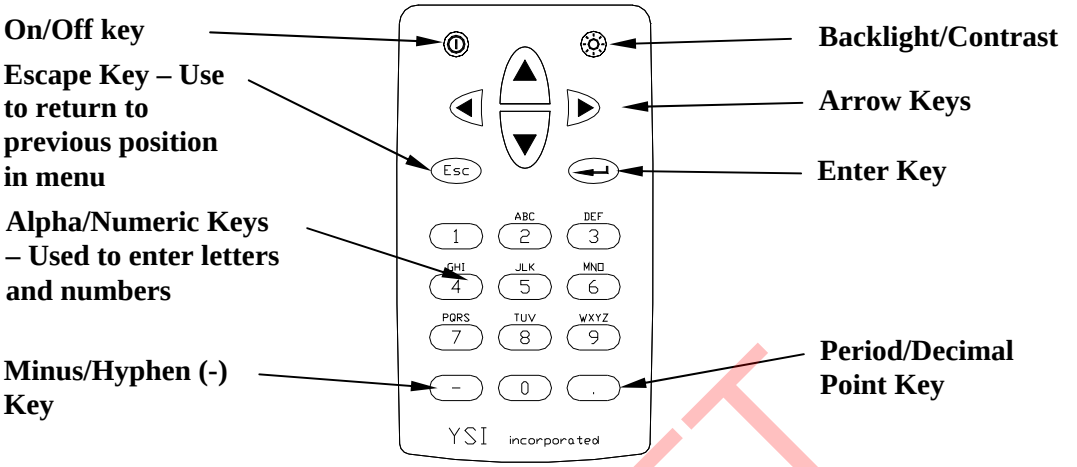

Figure 2.5 Keypad Features

KEY	LETTER/NUMBER
1	1
2	ABC2abc3
3	DEF3def3
4	GHI4ghi4
5	JKL5jkl5
6	MNO6mno6
7	PQRS7pqrs7
8	TUV8tuv8
9	WXYZ9wxyz9
0	0

Figure 2.6 Keypad Features

1. See Figure 2.10 Keypad Letters & Numbers and press the appropriate key repeatedly until letter or number desired appears in display.

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NOTE: Press the key repeatedly in rapid succession to get to the desired letter or number. If you pause for more than a second, the cursor automatically scrolls to the right to prepare for the next input.

EXAMPLE 1: Press the **6** key *once* and *release* to display an uppercase “M”.

EXAMPLE 2: Press the **6** key *four times* and *release* to display the number “6”.

EXAMPLE 3: Press the **6** key *five times* and *stop* to display a lowercase “m”.

2. Press the left arrow key to go back and reenter a number or setter that needs to be changed.

Press the **Enter** key when your entry is complete.

NOTE: The instrument software permits only numeric entries in many instances, such as when setting the clock or entering calibration parameters.

2.10 Instrument Reset

The YSI 556 MPS is characterized by sophisticated software that should provide trouble-free operation. However, as with all high-capability software packages, it is always possible that the user will encounter circumstances in which the instrument does not respond to keypad entry. If this occurs, the instrument function can easily be restored by removing and then reapplying battery power. Simply remove either your C-cells or rechargeable battery pack from the battery compartment, wait 30 seconds and then replace the batteries. See Section *2.4 Batteries* for battery removal/reinstallation instructions.

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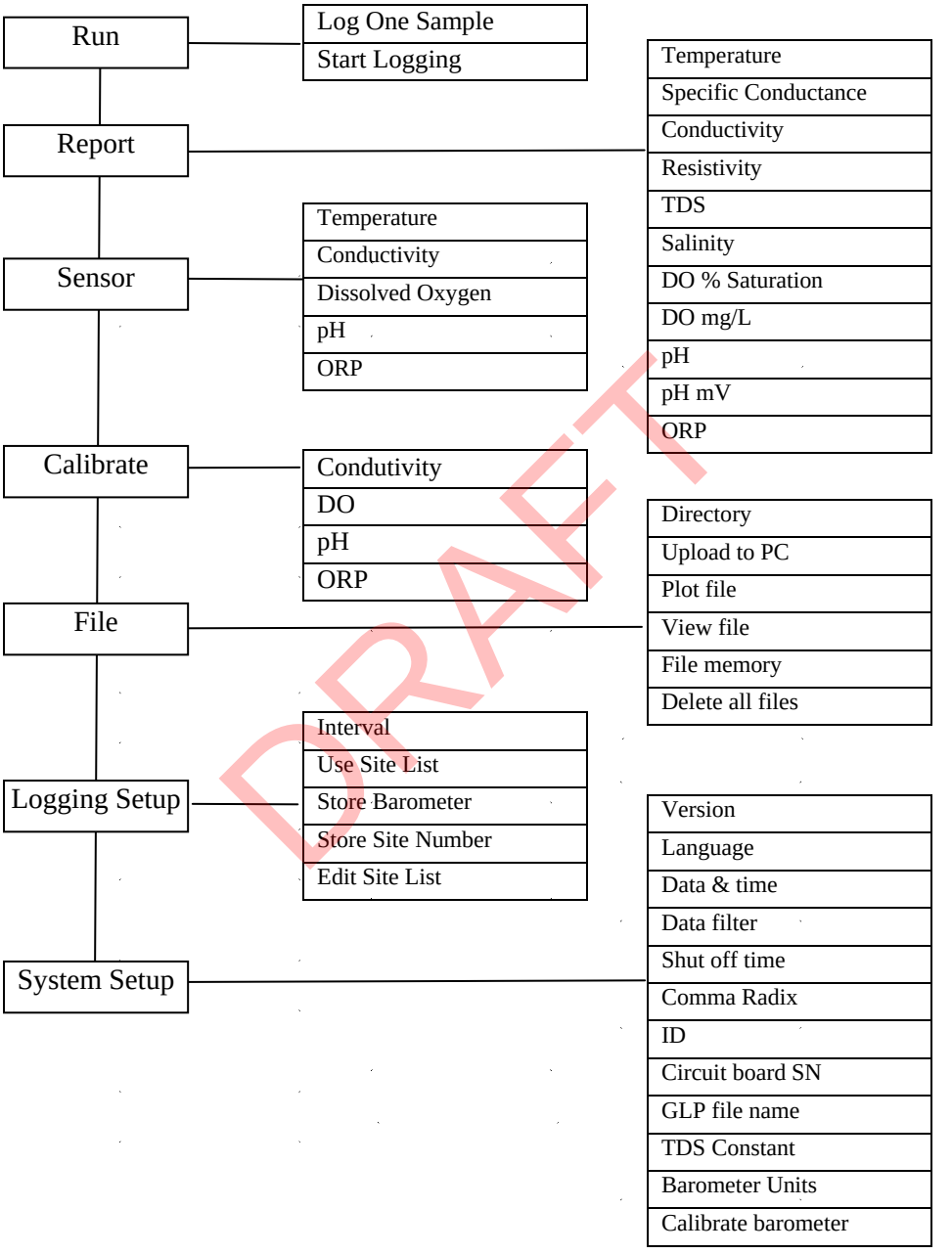
2.11 Menu Flowchart



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3. Probe Module

3.1 Introduction

The YSI 5563 Probe module is used for measuring dissolved oxygen, temperature, conductivity, and optional pH and ORP. The probe module is rugged, with the sensors enclosed in a heavy duty probe sensor guard with attached sinking weight. A 4, 10 or 20 meter cable is directly connected to the probe module body making it waterproof. An MS-19 connector at the end of the cable makes the YSI 5563 fully compatible with the YSI 556 Multi-Probe System.

3.2 Unpacking the Probe Module

1. Remove the YSI 5563 Probe Module from the shipping boxes.

   NOTE: Do not discard any parts or supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

NOTE: If any parts are missing or damaged, contact a YSI representative immediately. Refer to: *Appendix E Customer Service* o visit www.ysi.com.

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3.3 Features of the YSI 5563 Probe Module

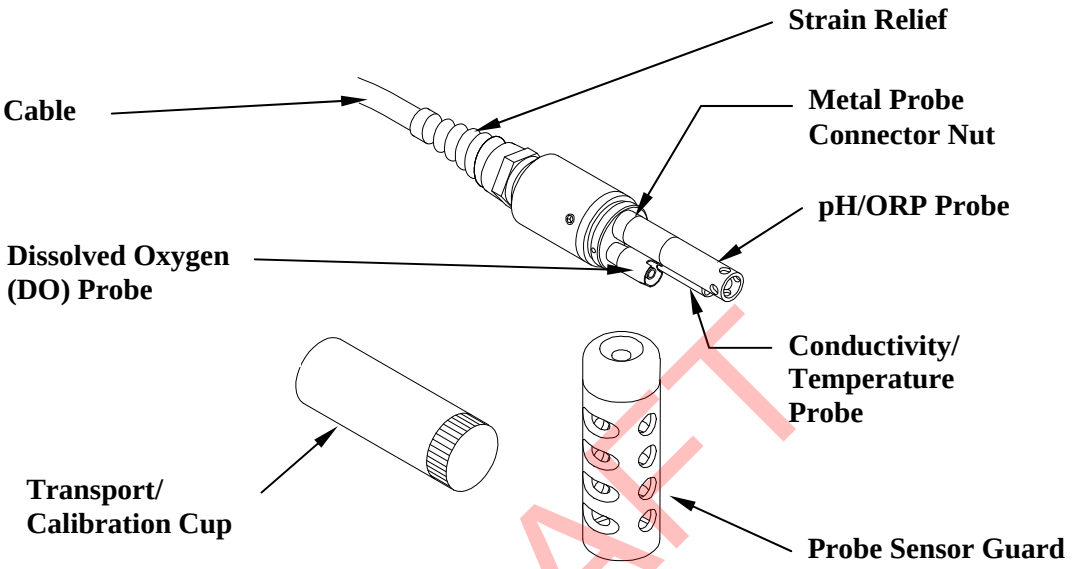

Figure 3.1 Probe Module

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3.4 Preparing the Probe Module

To prepare the probe module for calibration and operation, you need to install the sensors into the connectors on the probe module bulkhead. In addition to sensor installation, you need to install a new DO membrane cap.

3.4.1 Sensor Installation

Whenever you install, remove or replace a sensor, it is extremely important that the entire probe module and all sensors be thoroughly dried prior to the removal of a sensor or a sensor port plug. This will prevent water from entering the port. Once you remove a sensor or plug, examine the connector inside the probe module sensor port. If any moisture is present, use compressed air to completely dry the connector. If the connector is corroded, return the probe module to your YSI Distributor or directly to YSI Customer Service. Refer to *Appendix E Customer Service*.

Conductivity/Temperature and pH, pH/ORP Sensor Installation

1. Unscrew and remove the probe sensor guard.

2. Using the sensor installation tool supplied in the YSI 5511 maintenance kit, unscrew and remove the sensor port plugs.

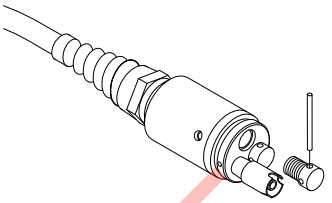

Figure 3.2 Port Plug Removal

3. Locate the port with the connector that corresponds to the sensor that is to be installed.

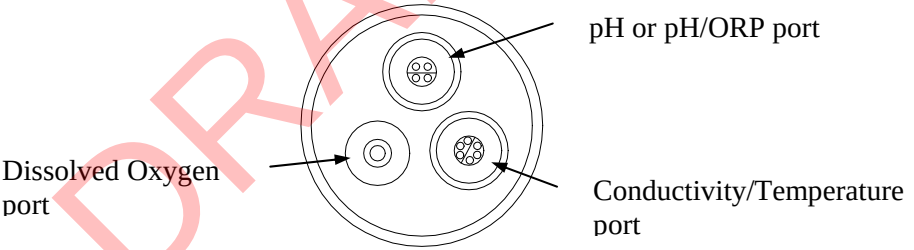

Figure 3.3 Sensor Port Identification

4. Apply a thin coat of o-ring lubricant (supplied in the YSI 5511 maintenance kit) to the o-rings on the connector side of the sensor (see Figure 3.4 O-ring Lubrication).

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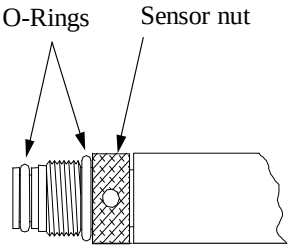

Figure 3.4 O-ring Lubrication

⚠ **CAUTION:** Make sure that there are NO contaminants between the o-ring and the sensor. Contaminants that are present under the o-ring may cause the o-ring to leak.

5. Be sure the probe module sensor port is free of moisture and then insert the sensor into the correct port. Gently rotate the sensor until the two connectors align.

6. With the connectors aligned, screw down the sensor nut using the sensor installation tool.

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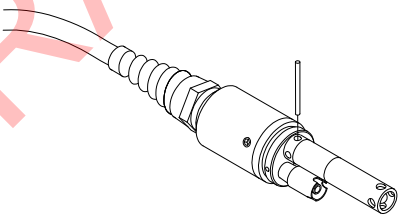

Figure 3.5 Sensor Installation

⚠ **CAUTION:** Do not cross thread the sensor nut. Tighten the nut until it is flush with the face of the probe module bulkhead. Do not over tighten.

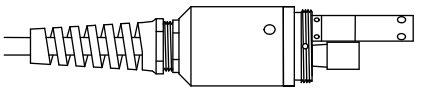

Figure 3.6 Bulkhead Seating

7. Repeat steps 3-6 for any other sensors.

8. Replace the probe sensor guard.

Dissolved Oxygen Sensor Installation

The YSI 5563 comes with the DO sensor already installed. Refer to Section *11.1.2 DO Sensor Replacement* for instructions on installing the YSI 559 Replaceable DO Module Kit.

3.4.2 Membrane Cap Selection

The YSI 5563 is shipped with a YSI 5909 kit that contains membrane caps made with 2 mil polyethylene (PE), a material which should be ideal for most field applications of the 556. However, YSI also offers membrane caps made with two other materials (1 mil polyethylene and 1 mil Teflon) which some users may also prefer. All membranes available for the 556/5563 system provide comparable accuracy if used properly. The difference between the two thicknesses of PE is found in the trade-off of flow dependence and response time as described below. Teflon is offered because some users may prefer to continue using the traditional membrane material used by YSI. To avoid confusion, the membrane caps are color coded as described below and can be ordered in kits as noted:

1 mil Teflon – Black Caps (Kit = YSI 5906)
1 mil Polyethylene (PE) – Yellow Caps (Kit = YSI 5908)
2 mil Polyethylene (PE) – Blue Caps (Kit = YSI 5909)

The 1 mil Teflon caps will offer traditional, reliable performance for most dissolved oxygen applications. The 1 mil PE caps will provide a significantly faster dissolved oxygen response (as long as your 556 Data Filter is set correctly as described below in Sections 10.2 and 10.3.1) while also giving readings which are significantly less flow dependent than the 1 mil Teflon caps. Finally, 2 mil PE caps will show a large reduction in flow dependence over 1 mil Teflon while not significantly increasing the response time. Generally, one of the PE caps is likely to provide better performance for your application.

IMPORTANT: No matter which type of membrane cap you select, you will have to confirm your selection in the 556 software from the Sensor menu as described in Section *4 Sensors*.

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3.4.3 Membrane Cap Installation

NOTE: The YSI 5563 DO sensor (already installed in the probe module) was shipped dry. A shipping membrane was installed to protect the electrode. **A new membrane cap must be installed before the first use.**

1. Unscrew and remove the probe sensor guard.
2. Unscrew, remove, and discard the old membrane cap.
3. Thoroughly rinse the sensor tip with distilled water.
4. Prepare the electrolyte according to the directions on the electrolyte solution bottle.
5. Hold the new membrane cap and fill it at least ½ full with the electrolyte solution.
6. Screw the membrane cap onto the sensor moderately tight. A small amount of electrolyte should overflow.

⚠ **Caution:** Do not touch the membrane surface.

7. Screw the probe sensor guard on moderately tight.

3.5 Transport/Calibration Cup

The YSI 5563 Probe module has been supplied with a convenient transport/calibration cup. This cup is an ideal container for calibration of the different sensors, minimizing the amount of solution needed. Refer to Section *6 Calibrate*.

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3.5.1 Transport/Calibration Cup Installation

1. Remove probe sensor guard, if already installed.

2. Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body.

3. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not over tighten as this could cause damage to the threaded portions.

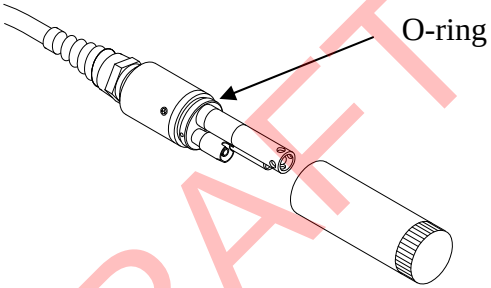

Figure 3.7 Transport/Calibration Cup Installation

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3.6 Instrument/Cable Connection

Attach the cable to the instrument as follows:

1. Line up the pins and guides on the cable with the holes and indentations on the cable connector at the bottom of the YSI 556 instrument. See Figure 2.1 Front View of YSI 556 MPS.

2. Holding the cable firmly against the cable connector, turn the locking mechanism clockwise until it snaps into place.

Remove the cable from the instrument by turning the cable connector counterclockwise until the cable disengages from the instrument.

4. Sensors

The Sensors screen allows the user to enable or disable each of the sensors and select which membrane material will be used for the dissolved oxygen sensor. Disabled sensors will not be displayed on the screen in real time or logged to files.

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

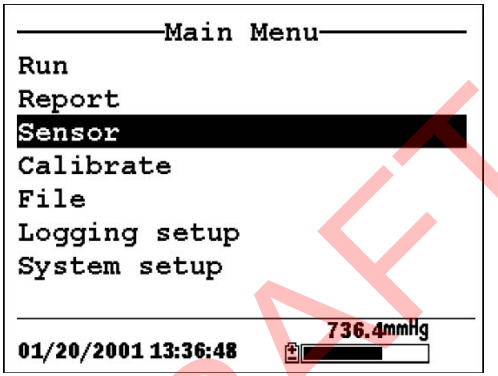

Figure 4.1 Main Menu Screen

3. Use the arrow keys to highlight the **Sensor** selection.

4. Press the **Enter** key to display the sensors enabled screen.

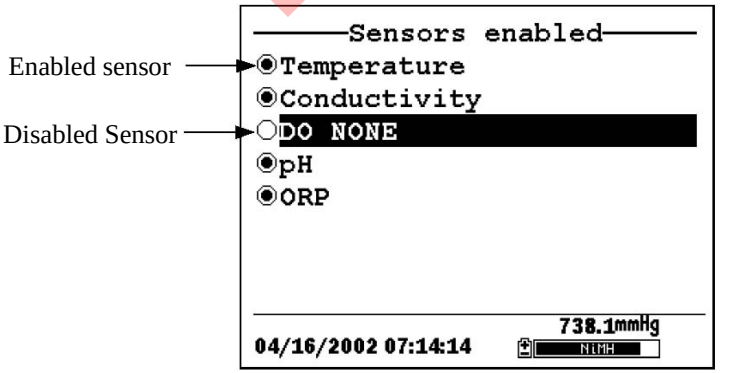

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Figure 4.2 Sensors Enabled Screen Before DO Membrane Selection

A black dot to the left of a sensor indicates that sensor is enabled. Sensors with an empty circle are disabled.

Highlight the "DO None" entry as shown above and press **Enter** to display the membrane choice screen. Consult Section *3.4.2 Membrane Cap Selection* for information on the advantages of each type of membrane material. Blue membrane caps using 2 mil polyethylene (PE) were shipped with your YSI 5563 and are likely to be the best choice for most 556 field applications.

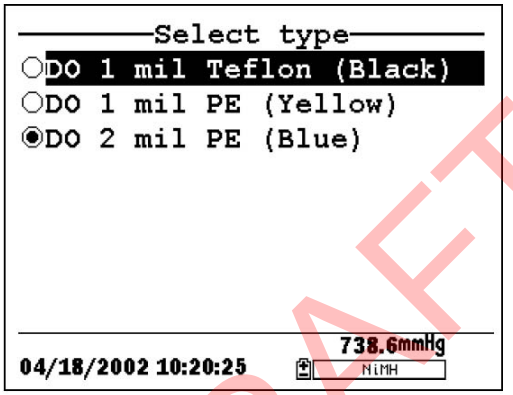

Figure 4.3 Membrane Selection Screen

Highlight the desired membrane choice – in this case, 2 mil PE - and press Enter to activate your selection with a dot to the left of the screen. Then press **Escape** to return to the Sensor menu that now shows your DO membrane selection.

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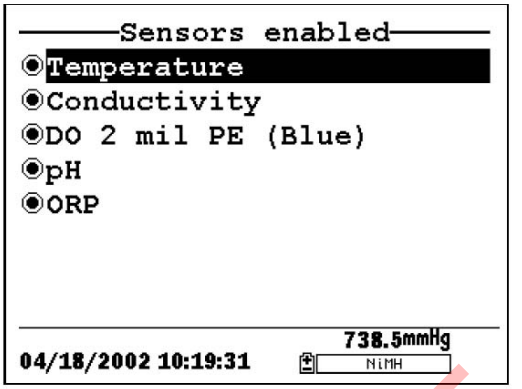

Figure 4.4 Sensors Enabled Screen After DO Membrane Selection

NOTE: The Temperature sensor cannot be disabled. Most other sensors require temperature compensation for accurate readings. In addition, the conductivity sensor must be activated in order to obtain accurate dissolved oxygen mg/L readings.

5. Use the arrow keys to highlight the sensor you want to change, then press the Enter key to enable or disable it.

6. Repeat step 5 for each sensor you want to change.

7. Press the Escape key to return to the main menu screen.

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5. Report

The Report Setup screen allows the user to select which sample parameters and units the YSI 556 MPS will display on the screen. It does NOT determine which parameters are logged to memory. Refer to Section *4 Sensors.*

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

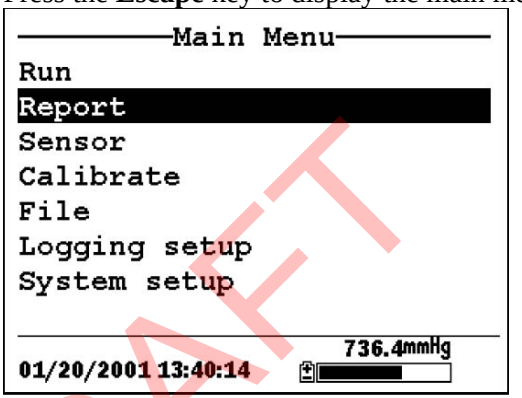

Figure 5.1 Main Menu

3. Use the arrow keys to highlight the **Report** selection.

4. Press the **Enter** key to display the report setup screen.

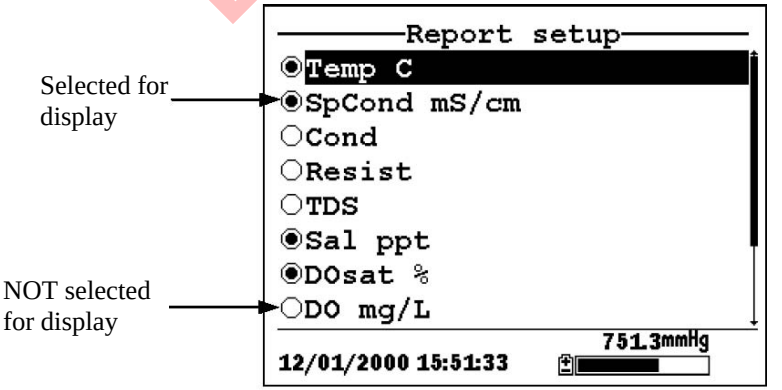

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Figure 5.2 Report Setup Screen

NOTE: A black dot to the left of a parameter indicates that parameter is selected for display. Parameters with an empty circle will not be displayed.

NOTE: You may have to scroll down past the bottom of the screen to see all the parameters.

5. Use the arrow keys to highlight the parameter you want to change, then press the **Enter** key. If you can't find the parameter you want, even after scrolling down past the bottom of the screen, the sensor used for that parameter is disabled. Refer to Section *4 Sensors*.

6. If you selected Temperature, Specific Conductivity, Conductivity, Resistance or Total Dissolved Solids, the Units screen will appear.

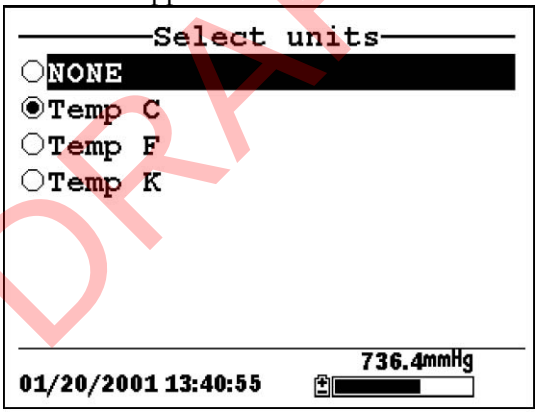

Figure 5.3 Units Screen

7. Use the arrow keys to select the units desired, then press the **Enter** key to return to the report setup screen.

8. Repeat steps 5 and 6 for each parameter you want to change.

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NOTE: Specific Conductance (temperature compensated conductivity) is notated on the Run screen with a small ‘c’ after the units of measure.

All parameters may be enabled at the same time.

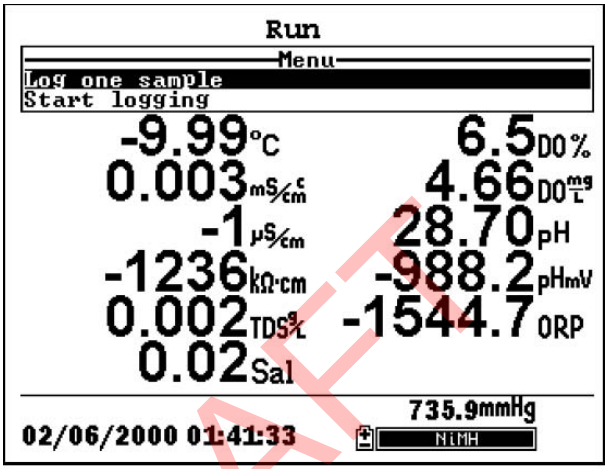

Figure 5.4 All Parameters Displayed

9. Press the **Escape** key to return to the Main menu screen.

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6. Calibrate

All of the sensors, except temperature, require periodic calibration to assure high performance. You will find specific calibration procedures for all sensors that require calibration in the following sections. If a sensor listed is not installed in your probe module, skip that section and proceed to the next sensor until the calibration is complete.

⚠ **CAUTION:** Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review *Appendix D Health and Safety*. Some calibration standard solutions may require special handling.

6.1 Getting Ready to Calibrate

6.1.1 Containers Needed to Calibrate the Probe Module

The transport/calibration cup that comes with your probe module serves as a calibration chamber for all calibrations and minimizes the volume of calibration reagents required.

Instead of the transport/calibration cup, you may use laboratory glassware to perform calibrations. If you do not use the transport/calibration cup that is designed for the probe module, you are cautioned to do the following:

- ✓ Perform all calibrations with the Probe Sensor Guard installed. This protects the sensors from possible physical damage.
- ✓ Use a ring stand and clamp to secure the probe module body to prevent the module from falling over. Most laboratory glassware has convex bottoms.
- ✓ Ensure that all sensors are immersed in calibration solutions. Many of the calibrations factor in readings from other sensors (e.g., temperature sensor). The top vent hole of the conductivity sensor must also be immersed during some calibrations.

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6.1.2 Calibration Tips

1. If you use the Transport/Calibration Cup for dissolved oxygen (DO) calibration, make certain to loosen the seal to allow pressure equilibration before calibration. The DO calibration is a water-saturated air calibration.

2. When calibrating pH, always calibrate with buffer 7 first, regardless if performing a 1, 2, or 3 point calibration

3. The key to successful calibration is to ensure that the sensors are completely submersed when calibration values are entered. Use recommended volumes when performing calibrations.

4. For maximum accuracy, use a small amount of previously used calibration solution to pre-rinse the probe module. You may wish to save old calibration standards for this purpose.

5. Fill a bucket with ambient temperature water to rinse the probe module between calibration solutions.

6. Have several clean, absorbent paper towels or cotton cloths available to dry the probe module between rinses and calibration solutions. Shake the excess rinse water off of the probe module, especially when the probe sensor guard is installed. Dry off the outside of the probe module and probe sensor guard. Making sure that the probe module is dry reduces carry-over contamination of calibrator solutions and increases the accuracy of the calibration.

7. If you are using laboratory glassware for calibration, you do not need to remove the probe sensor guard to rinse and dry the sensors between calibration solutions. The inaccuracy resulting from simply rinsing the sensor compartment and drying the outside of the guard is minimal.

8. If you are using laboratory glassware, remove the stainless steel weight from the bottom of the probe sensor guard by turning the weight counterclockwise. When the weight is removed, the calibration solutions have access to the sensors

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without displacing a lot of fluid. This also reduces the amount of liquid that is carried between calibrations.

9. Make certain that port plugs are installed in all ports where sensors are not installed. It is extremely important to keep these electrical connectors dry.

6.1.3 Recommended Volumes

Follow these instructions to use the transport/calibration cup for calibration procedures.

- ✓ Ensure that an o-ring is installed in the o-ring groove of the transport/calibration cup bottom cap, and that the bottom cap is securely tightened.

  NOTE: Do not over-tighten as this could cause damage to the threaded portions.

- ✓ Remove the probe sensor guard, if it is installed.
- ✓ Remove the o-ring, if installed, from the probe module and inspect the installed o-ring on the probe module for obvious defects and, if necessary, replace it with the extra o-ring supplied.
- ✓ Some calibrations can be accomplished with the probe module upright or upside down. A separate clamp and stand, such as a ring stand, is required to support the probe module in the upside down position.
- ✓ To calibrate, follow the procedures in the next section, Calibration Procedures. The approximate volumes of the reagents are specified below for both the upright and upside down orientations.
- ✓ When using the Transport/Calibration Cup for dissolved oxygen % saturation calibration, make certain that the vessel is vented to the atmosphere by loosening the bottom cap or cup assembly and that approximately 1/8 inch (3 cm) of water is present in the cup.

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Sensor to Calibrate	Upright	Upside Down
Conductivity	55ml	55ml
pH/ORP	30ml	60ml

Table 6.1 Calibration Volumes

6.2 Calibration Procedures

6.2.1 Accessing the Calibrate Screen

1. Press the **On/off** key to display the run screen.
2. Press the **Escape** key to display the main menu screen.
3. Use the arrow keys to highlight the **Calibrate** selection.

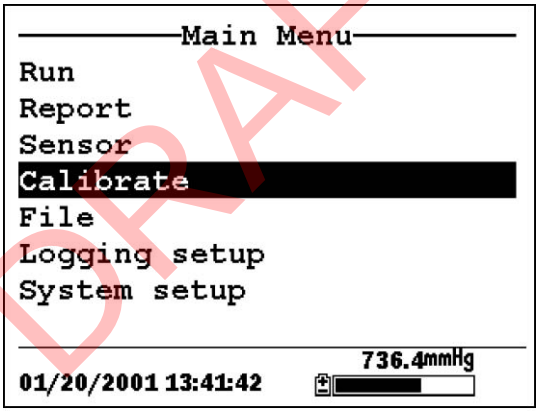

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Figure 6.1 Main Menu

4. Press the **Enter** key. The Calibrate screen will be displayed.

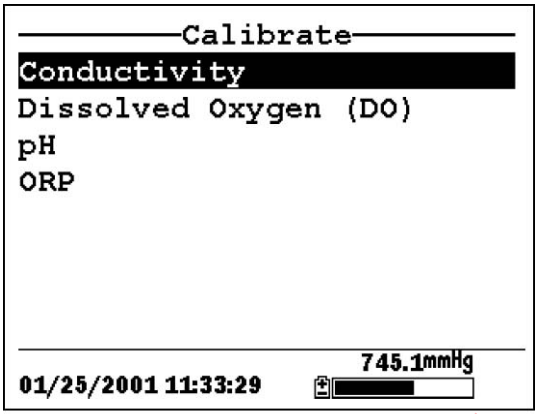

Figure 6.2 Calibrate Screen

6.2.2 Conductivity Calibration

This procedure calibrates specific conductance (recommended), conductivity and salinity. Calibrating any one option automatically calibrates the other two.

1. Go to the calibrate screen as described in Section *6.2.1Accessing the Calibrate Screen.*.
2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.
3. Press **Enter**. The Conductivity Calibration Screen is displayed.

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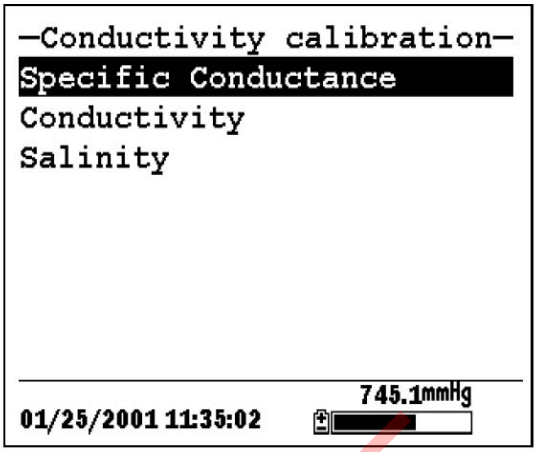

Figure 6.3 Conductivity Calibration Selection Screen

4. Use the arrow keys to highlight the Specific Conductance selection.

5. Press **Enter**. The Conductivity Calibration Entry Screen is displayed.

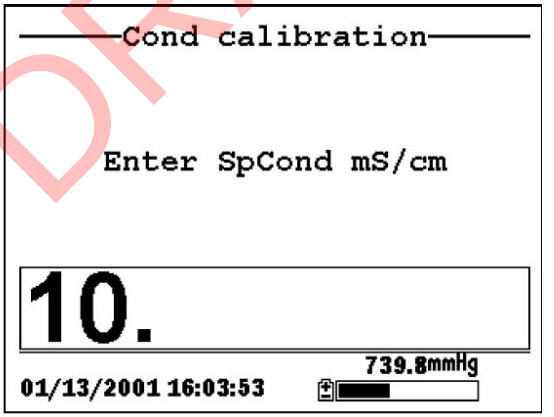

Figure 6.4 Conductivity Calibration Selection Screen

6. Place the correct amount of conductivity standard (see Table 6.1 Calibration Volumes) into a clean, dry or pre-rinsed transport/calibration cup.

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⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the conductivity standard you choose should be within the same conductivity range as the samples you are preparing to measure. However, we do not recommend using standards less than 1 mS/cm. For example:

- ✓ For fresh water use a 1 mS/cm conductivity standard.
- ✓ For brackish water use a 10 mS/cm conductivity standard.
- ✓ For seawater use a 50 mS/cm conductivity standard.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the conductivity sensor with a small amount of standard that can be discarded. Be certain that you avoid cross-contamination of solutions. Make certain that there are no salt deposits around the oxygen and pH/ORP sensors, particularly if you are employing standards of low conductivity.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the conductivity cell.

NOTE: The sensor must be completely immersed past its vent hole. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the vent hole is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

NOTE: Do not over tighten as this could cause damage to the threaded portions.

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10. Use the keypad to enter the calibration value of the standard you are using.

NOTE: Be sure to enter the value in **mS/cm at 25°C**.

11. Press **Enter**. The Conductivity Calibration Screen is displayed.

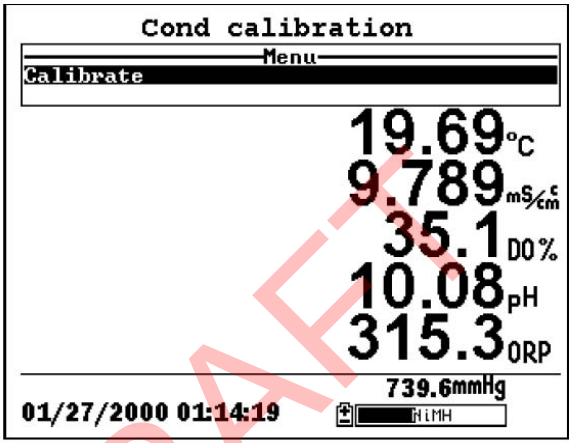

Figure 6.5 Conductivity Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

13. Observe the reading under Specific Conductance. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

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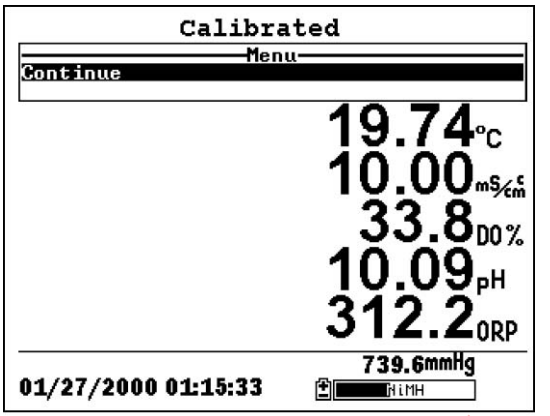

Figure 6.6 Calibrated

14. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen..

15. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen .

16. Rinse the probe module and sensors in tap or purified water and dry.

6.2.3 Dissolved Oxygen Calibration

This procedure calibrates dissolved oxygen. Calibrating any one option (% or mg/L) automatically calibrates the other.

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

NOTE: The instrument must be on for at least 10 - 15 minutes to polarize the DO sensor before calibrating.

2. Use the arrow keys to highlight the **Dissolved Oxygen** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The dissolved oxygen calibration screen is displayed.

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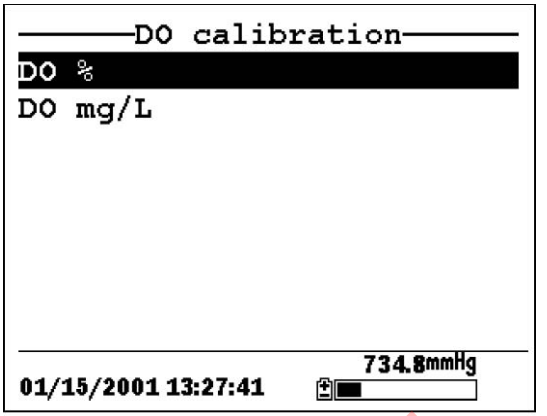

Figure 6.7 DO Calibration Screen

DO Calibration in % Saturation

1. Use the arrow keys to highlight the DO% selection.

2. Press **Enter**. The DO Barometric Pressure Entry Screen is displayed.

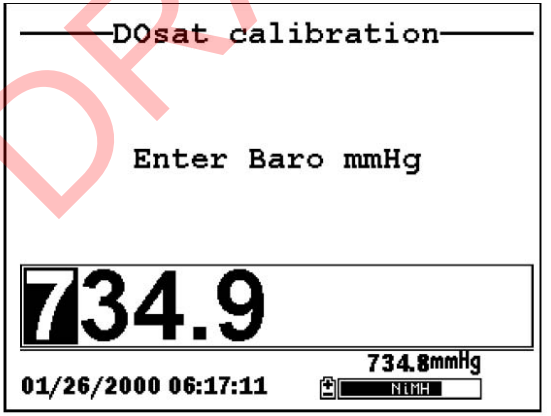

Figure 6.8 DO Barometric Pressure Entry Screen

3. Place approximately 3 mm (1/8 inch) of water in the bottom of the transport/calibration cup.

4. Place the probe module into the transport/calibration cup.

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NOTE: Make sure that the DO and temperature sensors are **not** immersed in the water.

5. Engage only 1 or 2 threads of the transport/calibration cup to ensure the DO sensor is vented to the atmosphere.

6. Use the keypad to enter the current local barometric pressure.

NOTE: If the unit has the optional barometer, no entry is required.

NOTE: Barometer readings that appear in meteorological reports are generally corrected to sea level and must be uncorrected before use (refer to Section *10.10 Calibrate Barometer, Step 2*).

7. Press **Enter**. The DO% saturation calibration screen is displayed.

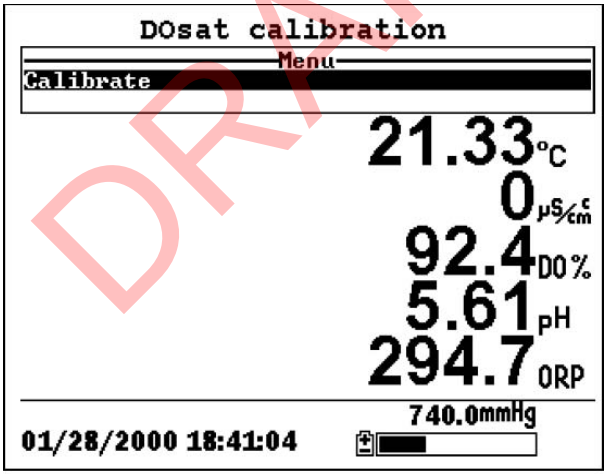

Figure 6.9 DO Sat Calibration Screen

8. Allow approximately ten minutes for the air in the transport/calibration cup to become water saturated and for the temperature to equilibrate before proceeding.

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9. Observe the reading under DO %. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue. See Figure 6.6 Calibrated.

10. Press **Enter**. This returns you to the DO calibration screen, See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

DO Calibration in mg/L

DO calibration in mg/L is carried out in a water sample which has a known concentration of dissolved oxygen (usually determined by a Winkler titration).

1. Go to the DO calibrate screen as described in Section *6.2.3 Dissolved Oxygen Calibration*, steps 1 through 3.

2. Use the arrow keys to highlight the **DO mg/L** selection.

3. Press **Enter**. The DO mg/L Entry Screen is displayed.

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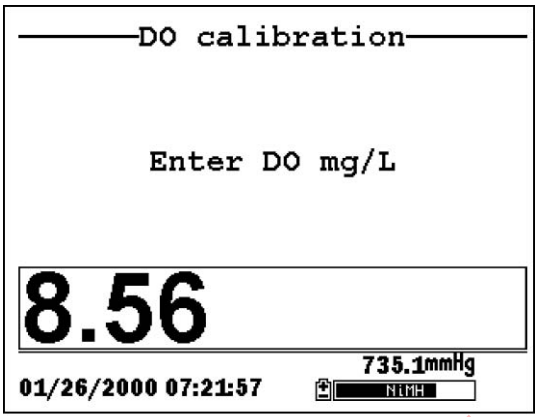

Figure 6.10 DO mg/L Entry Screen

4. Place the probe module in water with a known DO concentration.

NOTE: Be sure to completely immerse all the sensors.

5. Use the keypad to enter the known DO concentration of the water.

6. Press **Enter**. The Dissolved Oxygen mg/L Calibration Screen is displayed.

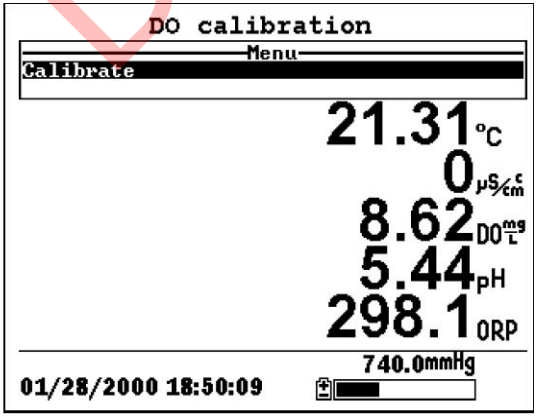

Figure 6.11 DO mg/L Calibration Screen

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7. Stir the water with a stir bar, or by rapidly moving the probe module, to provide fresh sample to the DO sensor.

8. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

9. Observe the DO mg/L reading, when the reading is stable (shows no significant change for approximately 30 seconds), press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

10. Press **Enter**. This returns you to the DO calibration screen. See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

6.2.4 pH Calibration

1. Go to the calibrate screen as described in *Section 6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **pH** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The pH calibration screen is displayed.

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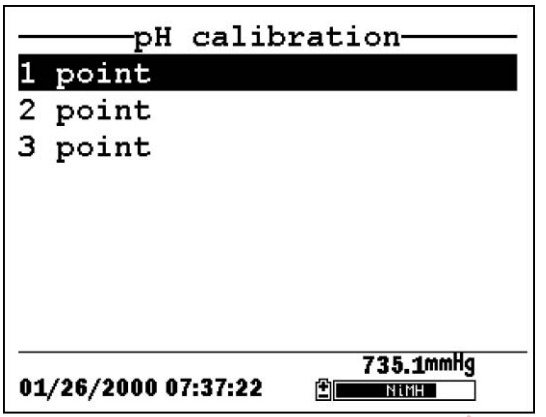

Figure 6.12 pH Calibration Screen

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- Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by carrying out a one point calibration. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select only one pH buffer.
- Select the **2-point** option to calibrate the pH sensor using only two calibration standards. Use this option if the media being monitored is known to be either basic or acidic. For example, if the pH of a pond is known to vary between 5.5 and 7, a two-point calibration with pH 7 and pH 4 buffers is sufficient. A three point calibration with an additional pH 10 buffer will not increase the accuracy of this measurement since the pH is not within this higher range.
- Select the **3-point** option to calibrate the pH sensor using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select a third pH buffer.

4. Use the arrow keys to highlight the **2-point** selection.

5. Press **Enter**. The pH Entry Screen is displayed.

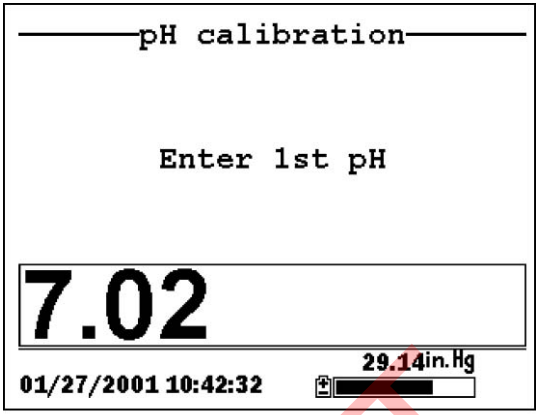

Figure 6.13 pH Entry Screen

6. Place the correct amount (see Table 6.1 Calibration Volumes) of pH buffer into a clean, dry or pre-rinsed transport/calibration cup.

NOTE: Always calibrate with buffer 7 first, regardless if performing a 1, 2, or 3 point calibration.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the pH buffers you choose should be within the same pH range as the water you are preparing to sample.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the pH sensor with a small amount of buffer that can be discarded. Be certain that you avoid cross-contamination of buffers with other solutions.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the pH sensor.

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NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the sensor is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten

NOTE: Do not over tighten as this could cause damage to the threaded portions.

10. Use the keypad to enter the calibration value of the buffer you are using **at the current temperature**.

NOTE: pH vs. temperature values are printed on the labels of all YSI pH buffers.

11. Press **Enter**. The pH calibration screen is displayed.

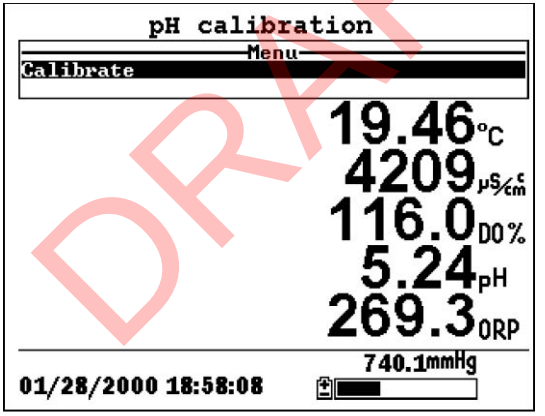

Figure 6.14 pH Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

13. Observe the reading under pH, when the reading shows no significant change for approximately 30 seconds, press **Enter**.

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The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

14. Press **Enter**. This returns you to the specified pH Calibration Screen, See Figure 6.13 pH Entry Screen.

15. Rinse the probe module, transport/calibration cup and sensors in tap or purified water and dry.

16. Repeat steps 6 through 13 above using a second pH buffer.

17. Press **Enter**. This returns you to the pH Calibration Screen, See Figure 6.12 pH Calibration Screen.

18. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

19. Rinse the probe module and sensors in tap or purified water and dry.

6.2.5 ORP Calibration

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **ORP** selection. See Figure 6.2 Calibrate Screen..

3. Press **Enter**. The ORP calibration screen is displayed.

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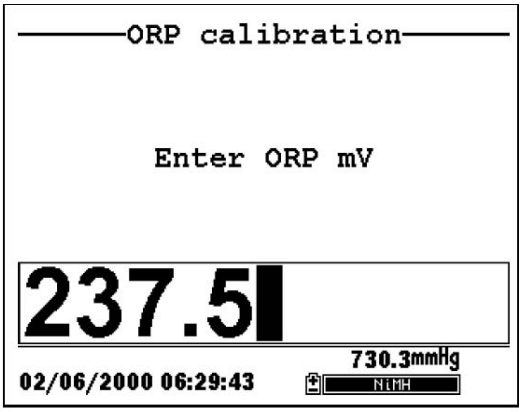

Figure 6.15 Specified ORP Calibration Screen

4. Place the correct amount (see Table 6.1 Calibration Volumes) of a known ORP solution (we recommend Zobell solution) into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the ORP sensor with a small amount of solution that can be discarded. Be certain that you avoid cross-contamination with other solutions.

5. Carefully immerse the sensor end of the probe module into the solution.

6. Gently rotate and/or move the probe module up and down to remove any bubbles from the ORP sensor.

NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes should ensure that the sensor is covered.

7. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

NOTE: Do not over tighten as this could cause damage to the threaded portions.

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8. Use the keypad to enter the correct value of the calibration solution you are using at the current temperature. Refer to Table 6.2 Zobell Solution Values.

Temperature °C	Zobell Solution Value, mV
-5	270.0
0	263.5
5	257.0
10	250.5
15	244.0
20	237.5
25	231.0
30	224.5
35	218.0
40	211.5
45	205.0
50	198.5

Table 6.2 Zobell Solution Values

9. Press **Enter**. The ORP calibration screen is displayed.

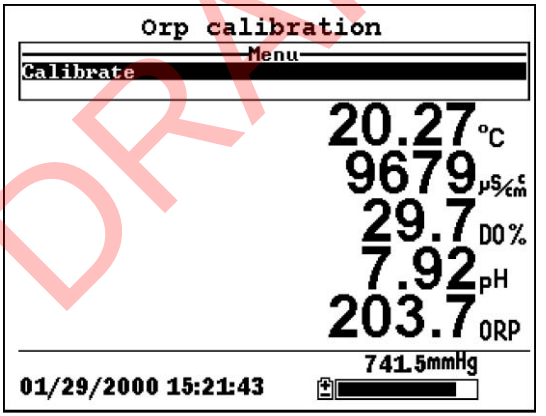

Figure 6.16 DO Cal Screen

10. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

NOTE: Verify that the temperature reading matches the value you used in Table 6.2 Zobell Solution Values.

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11. Observe the reading under ORP, when the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

12. Press **Enter**. This returns you to the Calibrate Screen. See Figure 6.2 Calibrate Screen.

13. Rinse the probe module and sensors in tap or purified water and dry.

6.3 Return to Factory Settings.

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen.*

2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.

NOTE: We will use the Conductivity sensor as an example; however, this process will work for any sensor.

3. Press **Enter.** The Conductivity Calibration Selection Screen is displayed. See Figure 6.3 Conductivity Calibration Selection Screen.

4. Use the arrow keys to highlight the **Specific Conductance** selection.

5. Press **Enter.** The Conductivity Calibration Entry Screen is displayed. See Figure 6.4 Conductivity Calibration Entry Screen.

6. Press and hold the **Enter** key down and press the **Escape** key.

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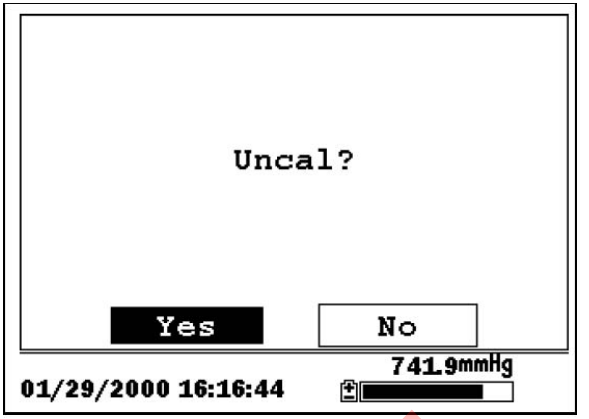

Figure 6.17 ORP Calibration Screen

7. Use the arrow keys to highlight the **YES** selection.

CAUTION: This returns a sensor to the factory settings. For example, in selecting to return specific conductance to the factory setting, salinity and conductivity will automatically return to their factory settings.

8. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen. .

9. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

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7. Run

The Run screen displays data from the sensors in real-time and allows the user to log sample data to memory for later analysis. Refer to Section *9 Logging* for details on logging sample data.

7.1 Real-Time Data

NOTE: Before measuring samples you must prepare the probe module (refer to Section *3.4 Preparing the Probe Module*), attach the probe module to the instrument (refer to Section *3.6 Instrument/Cable Connection*) and calibrate the sensors (refer to Section *6 Calibrate*).

1. Press the On/off key.

OR select Run from the main menu to display the run screen.

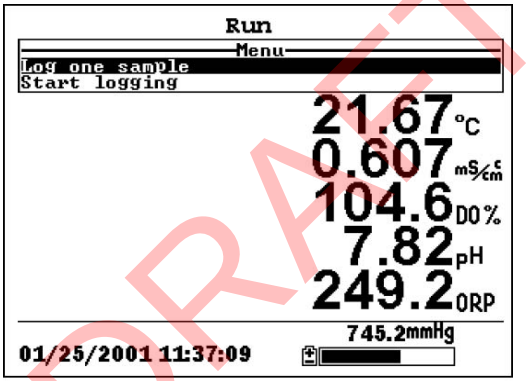

Figure 7.1 Run Screen

2. Make sure the probe sensor guard is installed.

3. Place the probe module in the sample. Be sure to completely immerse all the sensors.

4. Rapidly move the probe module through the sample to provide fresh sample to the DO sensor.

5. Watch the readings on the display until they are stable.

6. Refer to Section *9 Logging* for instructions on logging sample data.

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8. File

The File menu allows the user to view, upload or delete sample data and calibration record files stored in the YSI 556 MPS.

8.1 Accessing the File Screen

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

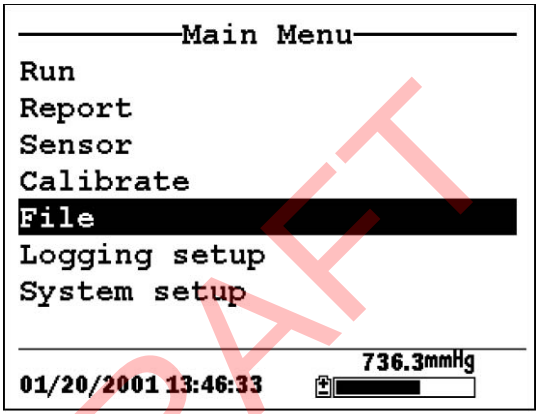

Figure 8.1 Main Menu Screen

3. Use the arrow keys to highlight the **File** selection.

4. Press the **Enter** key. The file screen is displayed.

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Figure 8.2 File Screen

8.2 Directory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.
2. Use the arrow keys to highlight the **Directory** selection. See Figure 8.2 File Screen.
3. Press the **Enter** key. The file list screen is displayed.

NOTE: Files are listed in the order in which they are logged to memory. Sample Data files have the file extension **.dat**, while Calibration Record files have the file extension **.glp**.

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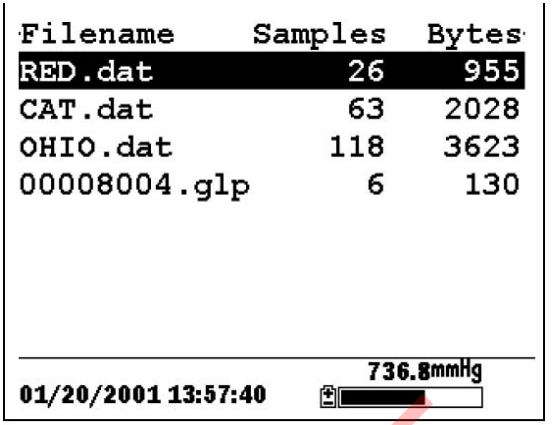

Figure 8.3 File List Screen

4. Use the arrow keys to highlight a file.

5. Press the **Enter** key. The file details screen is displayed.

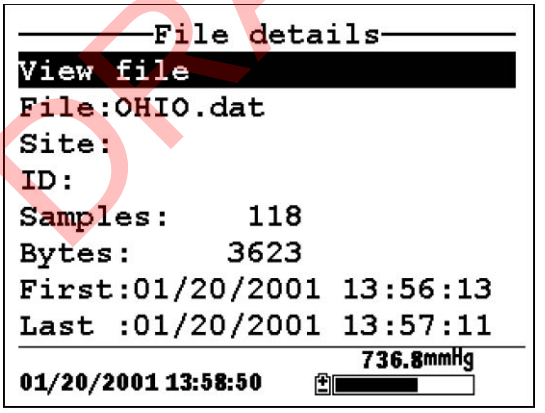

Figure 8.4 File Details Screen

6. Press the **Enter** key to view the file data. Refer to Section *8.3 View File* for details.

7. Press the **Escape** key repeatedly to return to the main menu screen.

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8.3 View File

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*. See Figure 8.2 File Screen.

2. Use the arrow keys to highlight the **View file** selection.

3. Press the **Enter** key. A list of files is displayed. See Figure 8.3 File List Screen.

4. Use the arrow keys to highlight an individual file.

   NOTE: You may have to scroll down to see all the files.

5. Press the **Enter** key. The file data is displayed with the file name at t8e top of the display.

   NOTE: If no file name was specified, the data is stored under the default name NONAME1.dat.

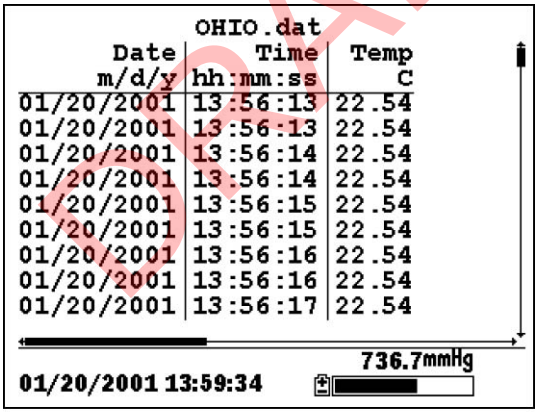

Figure 8.5 File Data Screen

6. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

7. Press the **Escape** key repeatedly to return to the main menu screen.

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8.4 Upload to PC

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. Refer to *Appendix G EcoWatch* for more information. EcoWatch for Windows® is available at no cost via a download from the YSI Web Site (www.ysi.com) or by contacting YSI Customer Support. Refer to *Appendix E Customer Service.*

8.4.1 Upload Setup

1. Disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

2. Connect the YSI 556 MPS to a serial (Comm) port of your computer via the 655173 PC Interface cable as shown in the following diagram:

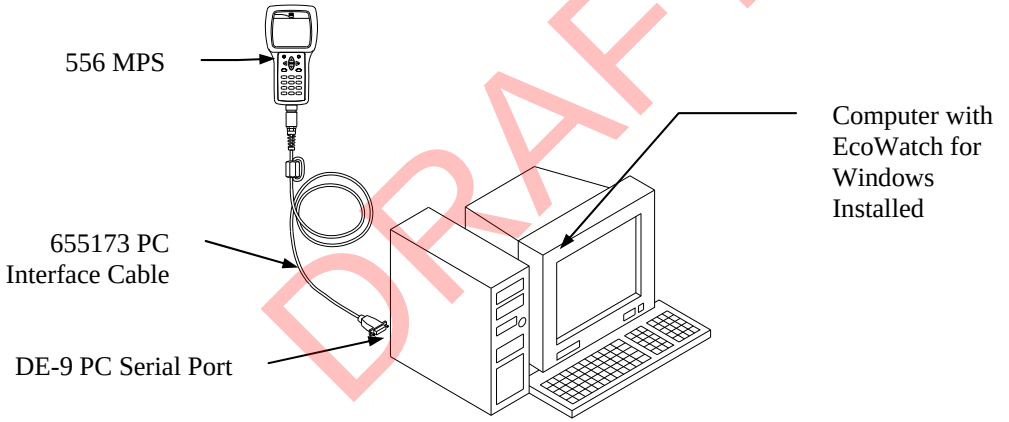

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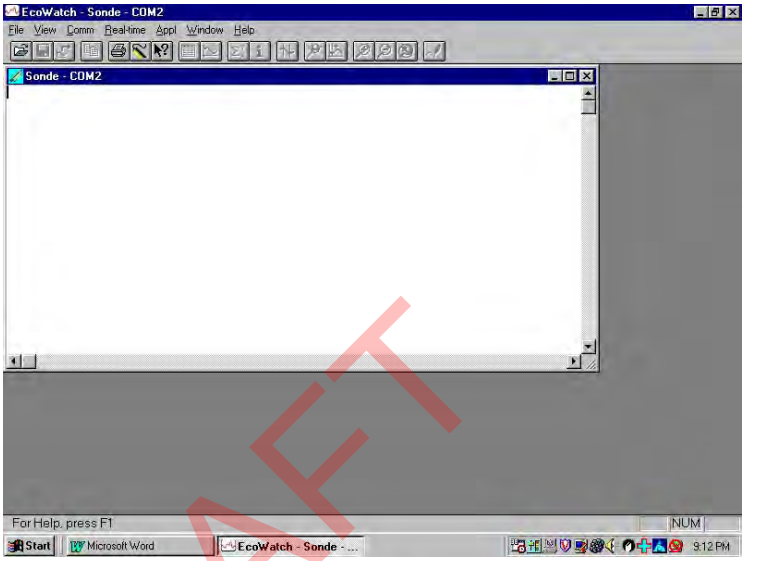
Figure 8.2 Computer/Instrument Interface

3. Open EcoWatch for Windows on your computer.

   NOTE: See *Appendix G EcoWatch* for installation instructions.

4. Click on the sonde/probe icon  in the upper toolbar.

5. Set the Comm port number to match the port the YSI 556 MPS is connected to. After this setup procedure, the following screen will be present on your PC monitor:



8.4.2 Uploading a .DAT File

1. Setup the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section *8.1 Accessing the File Screen.*

3. Use the arrow keys to highlight the **Upload to PC** selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the DAT file that you wish to transfer and press **Enter**, both the YSI 556 MPS and PC displays show the progress of the file transfer.

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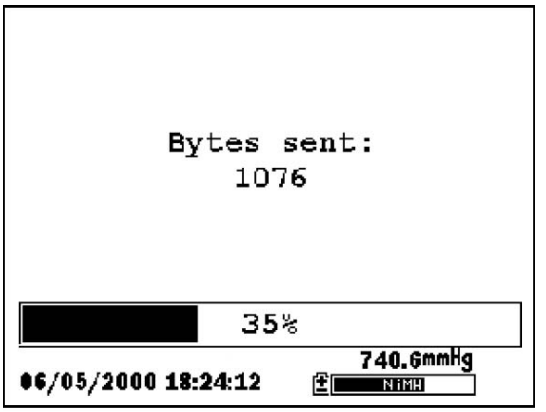
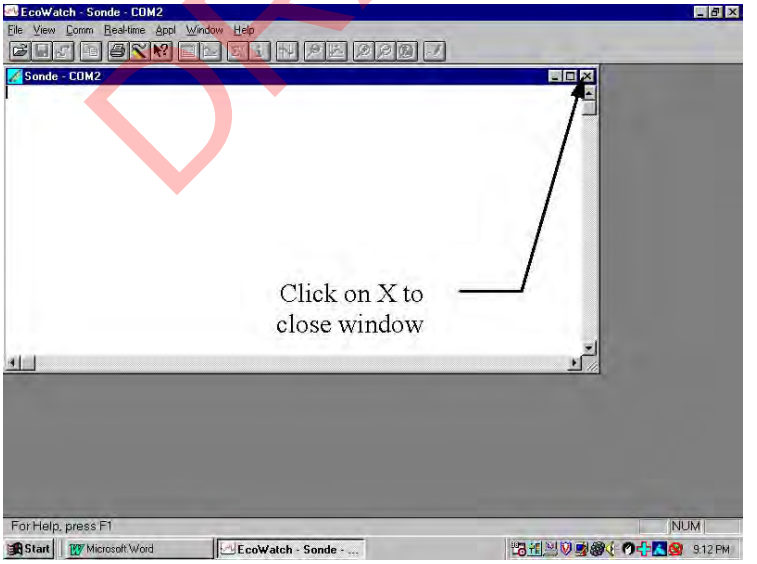


Figure 8.3 File Transfer Progress Screen

NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with a .DAT extension.

6. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the "X" at its upper right corner.



7. Press the **Escape** key on the YSI 556 MPS repeatedly to return

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to the main menu screen.

8.4.3 Uploading a Calibration Record (.glp) File

For more information on the calibration record, refer to *Appendix H Calibration Record Information*.

1. Setup up the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section

3. Use the arrow keys to highlight the Upload to PC selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the calibration record file that you wish to transfer and press **Enter.**

6. You will then be given a choice of uploading the file in three formats; **Binary, Comma & "" Delimited, and ASCII Text**.

   NOTE: The binary format is reserved for future YSI software packages.

7. Choose an option and press Enter, both the YSI 556 and PC displays show the progress of the file transfer.

   NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with the appropriate file extension.

   NOTE: To view the Calibration Record data after upload, simply open the .txt file in a general text editor such as Wordpad or Notepad.

8. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the "X" at its upper right corner.

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9. Press the **Escape** key repeatedly to return to the main menu screen.

8.5 File Memory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **File memory** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The file bytes used screen is displayed.

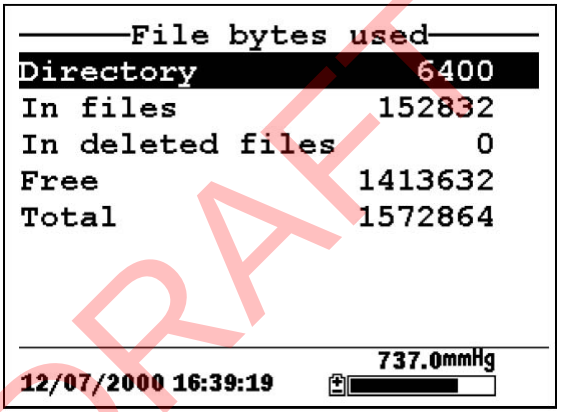

Figure 8.4 File Bytes Used Screen

4. The amount of free memory is listed in line 4 of the file bytes used screen.

   NOTE: If the amount of free memory is low, it may be time to delete all files (after first uploading all data to a PC). Refer to Section *8.6 Delete All Files*.

5. Press the **Escape** key repeatedly to return to the main menu screen.

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8.6 Delete All Files

NOTE: It is not possible to delete individual files in order to free up memory. The only way to free up memory is to delete ALL files present. Take care to transfer all files to your computer (refer to Section 8.4 Upload to PC) before deleting them.

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **Delete all files** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The Delete all Files screen is displayed.

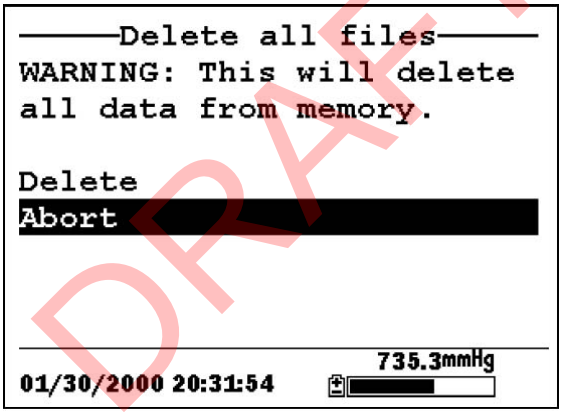

Figure 8.5 Delete All Files Screen

4. Use the arrow keys to highlight the **Delete** selection.

5. Press the **Enter** key.

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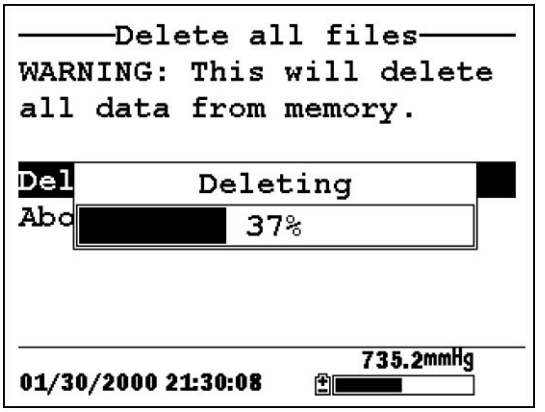

Figure 8.10 Deleting

The progress of file deletion is displayed in bar graph format.

NOTE: Deleting all files in the directory will not change any information in the site list.

6. Press the Escape key repeatedly to return to the main menu screen.

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9. Logging

9.1 Accessing the Logging Setup Screen

1. Press the **On/off** key to display the run screen.
2. Press the **Escape** key to display the main menu screen.

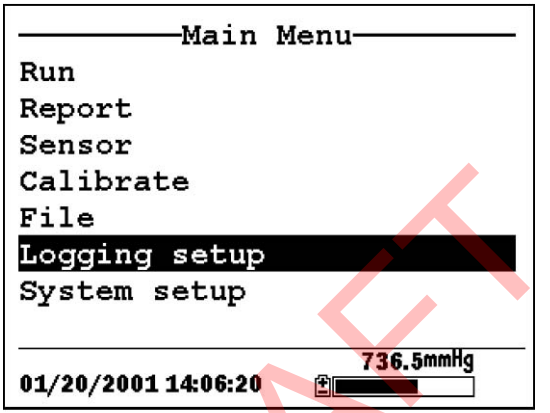

Figure 9.1 Main Menu

3. Use the arrow keys to highlight the **Logging setup** selection.
4. Press the **Enter** key. The logging setup screen is displayed.

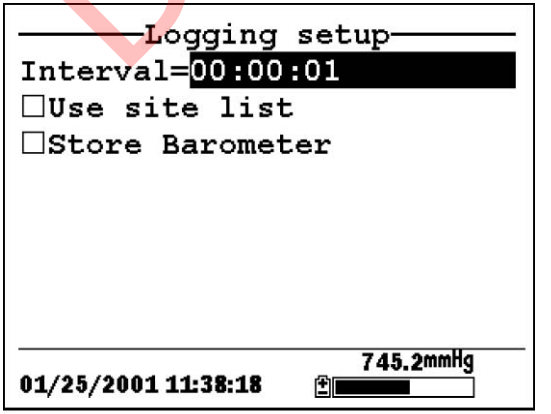

Figure 9.2 Setup Screen

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9.2 Setting Logging Interval

Follow steps below to set the interval for logging a data stream.

NOTE: If you do not specify an interval, the instrument will use a default interval setting of 1 second.

NOTE: It is not necessary to set a logging interval when logging a single sample.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the keypad to enter an interval between 1 second and 15 minutes. Refer to Section *2.9 Keypad Use*.

   NOTE: The interval field has hour, minute and second entry fields. Any entry over 1 hour will change automatically to a 15-minute setting.

3. Press the **Enter** key. The data stream interval is set.

4. Press the **Escape** key repeatedly to return to the main menu screen.

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9.3 Storing Barometer Readings

NOTE: The **Store barometer** option is only available on instruments that are equipped with the optional barometer.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Store barometer** selection. See Figure 9.2 Logging Setup Screen.

3. Press the **Enter** key until a check mark is entered in the box next to the store barometer selection if you want to log barometric readings.

OR press the **Enter** key until the box next to the barometer selection is empty if you do not want to log barometric readings.

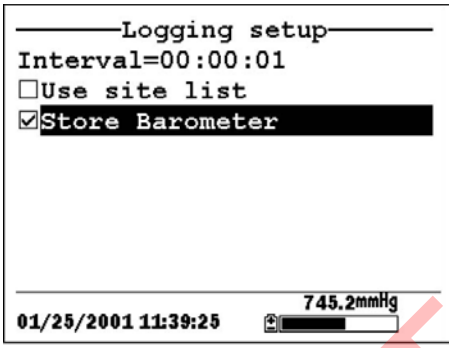

Figure 9.3 Store Barometer

4. Press the **Escape** key repeatedly to return to the main menu screen.

9.4 Creating a Site List

The site list option allows you to define file and site descriptions in the office or laboratory before moving to field logging studies. This is usually more convenient than entering the information at the site and is particularly valuable if you are visiting certain sites on a regular basis. The following section describes how to set up site lists which contain entries designated "Site Descriptions" that will be instantly available to the user in the field to facilitate the logging of data with pre-established naming of files and sites. There are two kinds of **Site Descriptions** available for use in Site lists:

• **Site Descriptions** associated with applications where data from a single site is always logged to a single file. This type is referred to as a "Single-Site Description" and is characterized by two parameters – a file name and a site name. Files logged to YSI 556 MPS memory under a **Single-Site Description** will be characterized primarily by the file name, but will also have the Site name attached, so that it is viewable in either the YSI 556 MPS **File directory** or in EcoWatch for Windows after upload to a PC

• **Site Descriptions** associated with applications where data from multiple sites are logged to a single file. This type is referred to as a "Multi-site Description" and is characterized by three parameters – a file name, a site name, and a site number. Files logged to YSI 556 MPS memory under a

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Multi-site Description are characterized by a file name, but not a site name, since multiple sites are involved. However, each data point has a Site Number attached to it so that the user can easily determine the sampling site when viewing the data from the YSI 556 MPS **File** menu or processing the data in EcoWatch for Windows after upload to a PC.

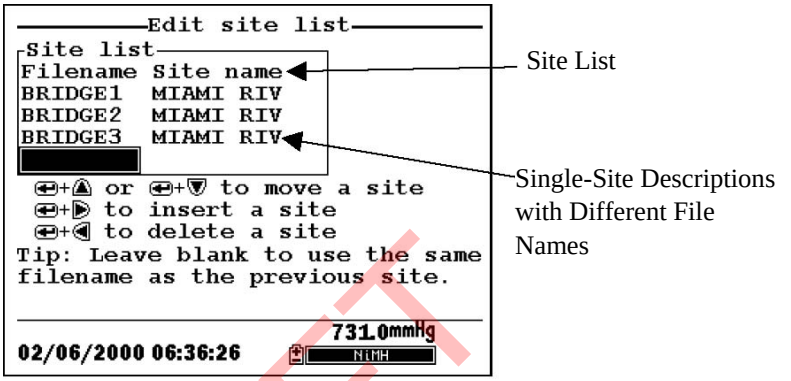

Figure 9.4 Single-Site Descriptions

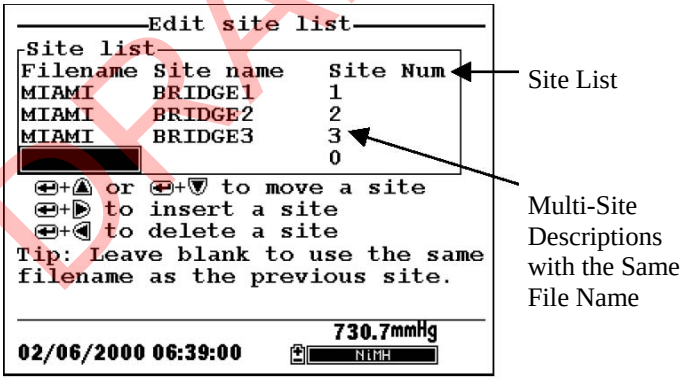

Figure 9.5 Multiple-Site Descriptions

NOTE: Site lists containing Single Site Descriptions are usually input with the designation **Store Site Number** INACTIVE in the YSI 556 MPS **Logging setup** menu. Thus, no site numbers appear in the first **Site list** example. Conversely, **Site lists** containing **Multi-Site Descriptions** MUST be input with the **Store Site Number** selection ACTIVE as shown in the second example.

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To create a site list:

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Use site list** selection.

3. Press the **Enter** key. A check mark is entered in the box next to the use site list selection *and* two new entries appear on the logging setup screen. See Figure 9.6 Logging Setup Screen.

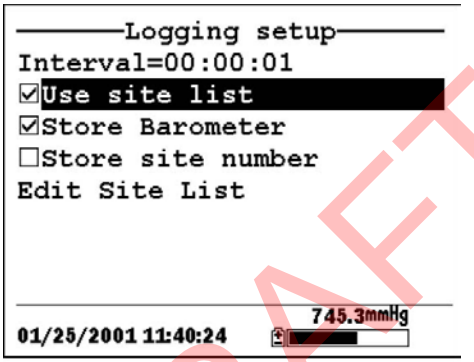

Figure 9.6 Logging Setup Screen

4. Use the arrow keys to highlight the **Store site number** selection.

5. If you are creating Multi-Site Descriptions (which require that the site **number** be stored in your data files), press the **Enter** key until a check mark appears in the box next to the store site number selection.

OR Press the **Enter** key until the box next to the store site number selection is empty, to create Single-Site Descriptions. The site **name** will be stored in the header of your data files.

6. Use the arrow keys to highlight the **Edit site list** selection.

7. Press the **Enter** key. The edit site list screen is displayed. See Figure 9.7 Edit Site List Screen. The **Filename** field is ready for input.

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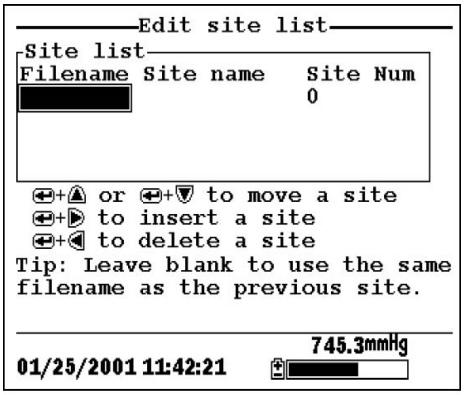

Figure 9.7 Edit Site List Screen

8. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.

9. Press the **Enter** key. The cursor moves to the right for the entry of a **Site name**.

10. Use the keypad to enter a site name up to 11 characters in length. Refer to Section *2.9 Keypad Use*.

NOTE: If the store site number selection is *not* checked, skip to Step 13.

11. Press the **Enter** key. The cursor moves to the site number entry position.

12. Use the keypad to enter a site number up to 7 characters in length. Refer to Section *2.9 Keypad Use*.

13. Press **Enter**. The cursor moves to the next filename entry position.

14. Repeat Steps 8 to 13 until all filenames and sites have been entered.

15. Press **Escape** repeatedly to return to the main menu screen.

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9.5 Editing a Site List

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Edit Site List** selection. See Figure 9.6 Logging Setup Screen.

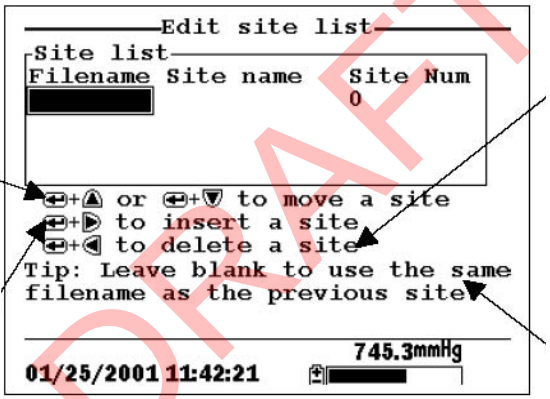
3. Press the **Enter** key. The edit site list screen is displayed.

4. Edit the site list using the keystrokes described below.

NOTE: Editing the site list will not have any effect on files stored in the instrument memory.

To MOVE a site:
Use the arrow keys to highlight a site. Press the Up or Down arrow key while holding down the Enter key.

To INSERT a site above another site:
Use the arrow keys to highlight the site. Press the Right arrow key while holding down the Enter key. Use keypad to input letters. Refer to Section 2.9 *Keypad Use.*



To DELETE a site:
Use the arrow keys to highlight a site. Press the Left arrow key while holding down the Enter key.

To use the same file name as the previous site: **Leave the filename blank**

Figure 9.1 Keystrokes for Editing Site List

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9.6 Logging Data Without a Site List

1. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

2. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged.

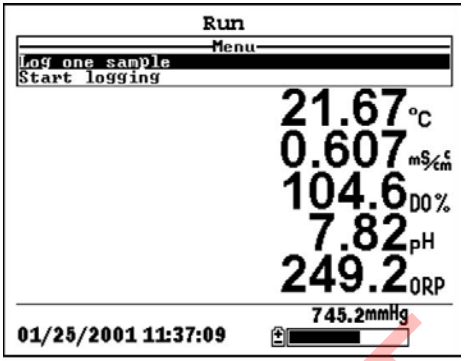

Figure 9.9 Run Screen

3. Press the **Enter** key. The Enter information screen is displayed.

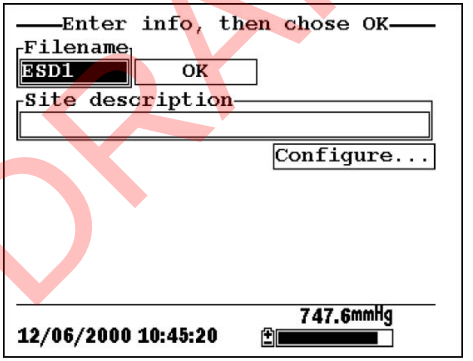

Figure 9.10 Enter Information Screen

NOTE: The last filename used will be displayed.

4. Use the keypad to enter a file name. Refer to Section *2.9 Keypad Use*.

NOTE: The instrument will assign a default file name of NONAME if no file name is specified.

5. Press the **Enter** key to input the file name.

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6. Use the arrow keys to highlight the **Site description** field in the enter information screen.

   NOTE: Entering a Site Description is optional. You may leave the Site Description blank and skip to Step 9.

7. Use the keypad to enter a site description name. Refer to Section *2.9 Keypad Use*.

8. Press the **Enter** key to input the site description.

   NOTE: If you want to change the logging setup, such as sampling interval or storing the barometer reading, use the arrow keys to highlight the **Configure** field, press the **Enter** key, then refer to Section *9.2 Setting Logging Interval* or *9.3 Storing Barometer Readings* for details.

9. Use the arrow keys to highlight the **OK** field in the center of the information screen.

10. Press the **Enter** key to start logging.

    NOTE: If the parameter mismatch screen is displayed, refer to Section *9.8 Adding Data to Existing Files*.

11. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. Skip to Step

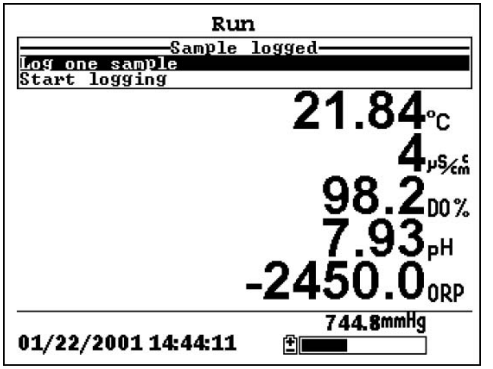

Figure 9.11 Sample Logged Screen

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If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**.

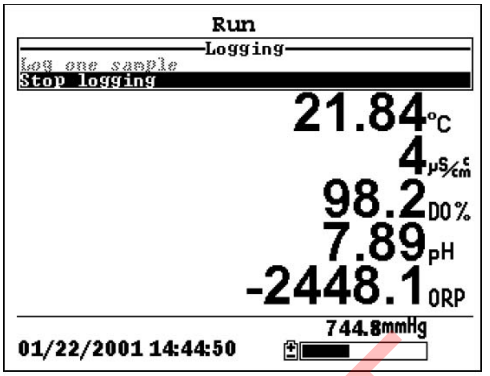

Figure 9.12 Logging Screen

12. At the end of the logging interval, press **Enter** to stop logging.

13. Refer to Section *8.3 View File* to view the data on the instrument display.

9.7 Logging Data with a Site List

1. If you have not already created a site list, refer to Section *9.4 Creating a Site List*.

2. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

3. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged. See Figure 9.9 Run Screen.

4. Press the **Enter** key. The Pick a site screen is displayed.

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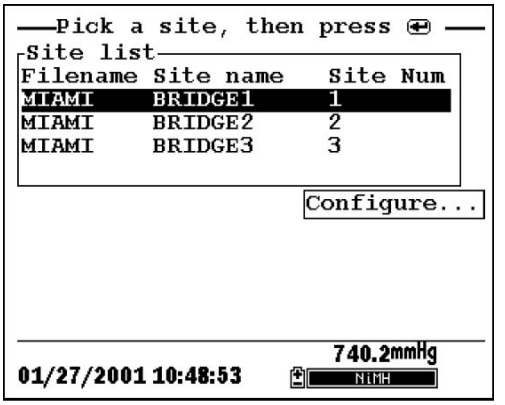

Figure 9.13 Pick a Site Screen

5. Use the arrow keys to highlight the **site** of your choice.

   NOTE: If the site of your choice is grayed out in the site list, refer to Section *9.8 Adding Data to Existing Files.*

   NOTE: Refer to Section *9.5 Editing a Site List* if you want to edit the site list.

6. Press the **Enter** key to start logging.

   NOTE: If the parameter mismatch screen is displayed, refer to Section 9.8 Adding Data to Existing Files.

7. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. See Figure 9.11 Sample Logged Screen. Skip to Step 9.

   If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**. See Figure 9.12 Logging Screen.

8. At the end of the logging interval, press **Enter** to stop logging.

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9. Refer to Section *8.3 View File* to view the data on the instrument display.

9.8 Adding Data to Existing Files

In order to add new data to an existing file, the current logging and sensor setup must be *exactly* the same as when the file was created. The following settings must be the same:

- **Sensors enabled** (refer to Section *4 Sensors*)
- **Store Barometer** (refer to Section *9.3 Storing Barometer Readings*)
- **Store Site Number** (refer to Section *9.4 Creating a Site List*)

If the current logging setup is not exactly the same as when the file was created, a parameter mismatch screen is displayed.

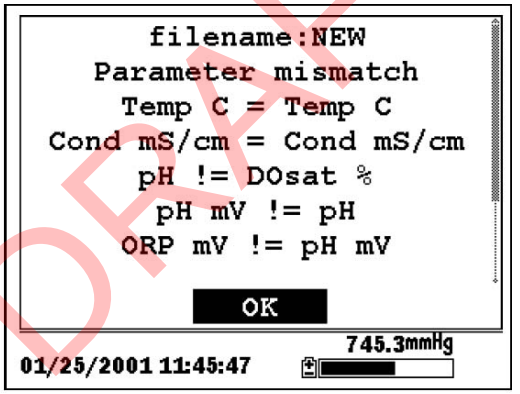

Figure 9.14 Parameter Mismatch Screen

NOTE: The right column shows parameters used when the file was created. The left column shows current parameters.

1. Press the **Down Arrow** key to scroll down and find the mismatch(es).

2. Use the following chart to resolve the mismatch(es).

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Mismatch	Action	Reference
Sensor(s) missing from left column	Enable the missing sensor(s)	Section *4 Sensors*
Extra sensor(s) listed in left column	Disable the extra sensor(s)	Section *4 Sensors*
Barometer missing from left column, but present in right column	Enable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Barometer present in left column, but missing from right column	Disable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Store Site Number missing from left column, but present in right column	Enable the Store Site Number setting	Section *9.4 Creating a Site List*
Store Site Number present in left column, but missing from right column	Disable the Store Site Number setting	Section *9.4 Creating a Site List*

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3. Return to Section *9.6 Logging Data without a Site List* or *9.7 Logging Data with a Site List*.

10. System Setup

The YSI 556 MPS has a number of features that are user-selectable or can be configured to meet the user's preferences. Most of these choices are found in the **System setup** menu.

10.1 Accessing the System Setup Screen

1. Press the **On/off** key to display the run screen. See Figure Front View of YSI 556 MPS.

2. Press the **Escape** key to display the main menu screen.

3. Use the arrow keys to highlight the **System setup** selection.

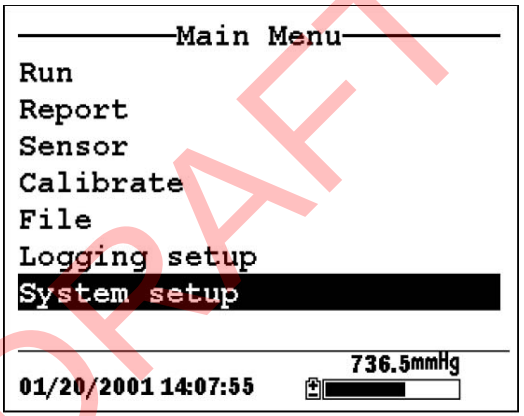

Figure 10.1 Main Menu

4. Press the **Enter** key. The system setup screen is displayed.

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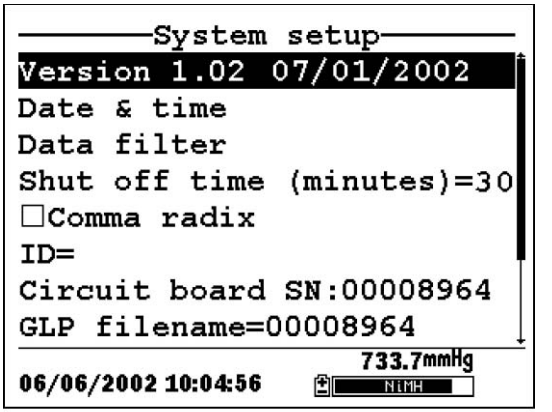

Figure 10.2 System Setup Screen

NOTE: The first line of the **System setup** menu shows the current software version of your YSI 556 MPS. As software enhancements are introduced, you will be able to upgrade your YSI 556 MPS from the YSI Web site. Refer to Section *11.2 Upgrading YSI 556 MPS* Software for details.

10.2 Language Setting

1. Go to the System Setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight **Language** on the System Setup screen. Press **Enter** to open the Language screen. .
3. Use the arrow keys to highlight your desired **Language**. Press **Enter**.
4. Press the Escape key repeatedly to return to the Main men

10.3 Date and Time Setup

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

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2. Use the arrow keys to highlight the **Date & time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press **Enter**. The date and time setup screen is displayed.

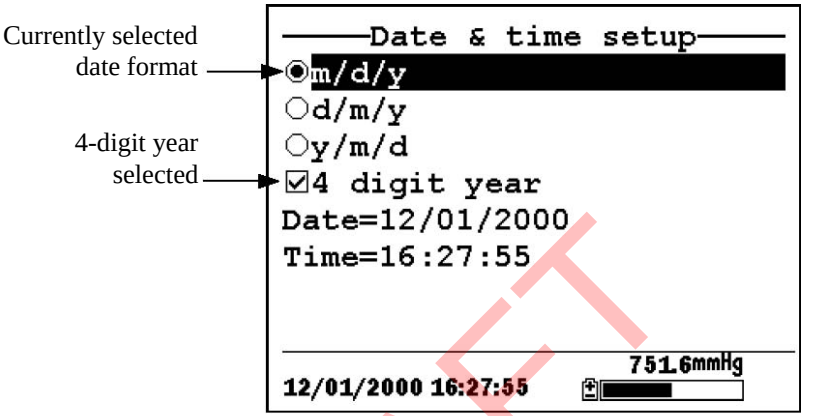

Figure 10.3 Date Setup Screen

NOTE: A black dot to the left of a date format indicates that format is selected.

4. Use the arrow keys to highlight your desired date format.

5. Press **Enter**.

6. Use the arrow keys to highlight the 4-digit year selection.

7. Press **Enter**. A check mark appears in the check box next to the 4-digit year selection.

   NOTE: If unchecked, a 2-digit year is used.

8. Use the arrow keys to highlight the **Date** selection.

9. Press **Enter**. A cursor appears over the first number in the date.

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10. Enter the proper number from the keypad for the highlighted date digit. The cursor moves automatically to the next date digit. Refer to Section *2.9 Keypad Use* for more keypad information.

11. Repeat Step 10 until all date digits are correct.

12. Press **Enter** to input the specified date.

13. Use the arrow keys to highlight the **Time** selection.

14. Press **Enter**. A cursor appears over the first number in the time selection.

15. Enter the proper number from the keypad for the highlighted time digit. The cursor moves automatically to the next time digit.

    NOTE: Use military format when entering time. For example, 2:00 PM is entered as 14:00.

16. Repeat Step 15 until all time digits are correct.

17. Press **Enter** to input the correct time.

18. Press the **Escape** key repeatedly to return to the Main menu screen.

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10.4 Data Filter

The Data Filter is a software filter that eliminates sensor noise and provides more stable readings.

NOTE: YSI recommends using the default values for the data filter for most field applications.

However, users who are primarily interested in a fast response from their dissolved oxygen sensor should consider a change of the default time constant setting of 8 seconds to one of 2 seconds. This change can be made according to the instructions in Section *10.3.1 Changing the Data Filter Settings* below. The disadvantage of lowering the time constant is that field pH readings may appear somewhat noisy if the cable is in motion.

10.4.1 Changing the Data Filter Settings

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Data filter** selection. See Figure 10.1 Main Menu.

3. Press the **Enter** key. The Data filter setup screen is displayed.

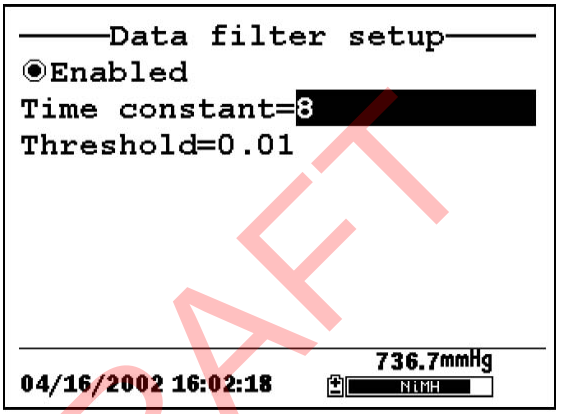

Figure 10.4 Data Filter Screen

4. With Enabled highlighted, press the **Enter** key to Enable or Disable the data filter. A black dot to the left of the selection indicates the data filter is enabled.

5. Use the arrow keys to highlight the **Time constant** field.

   NOTE: This value is the time constant in seconds for the software data filter. Increasing the time constant will result in greater filtering of the data, but will also slow down the apparent response of the sensors.

6. Use the keypad to enter a value. The default value is 8 and this value is ideal for most 556 field applications. As described in Section *10.3 Data Filter* above, users who wish to decrease the response time of the DO readings at the expense of some noise for the pH readings determined

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concurrently, should change the Time Constant to a value of 2.

7. Press the **Enter** key to enter the time constant.

8. Use the arrow keys to highlight the **Threshold** field.

   NOTE: This value determines when the software data filter will engage/disengage, speeding the response to large changes in a reading. When the difference between two consecutive readings is larger than the threshold, then the reading is displayed unfiltered. When the difference between two consecutive readings drops below the threshold, readings will be filtered again.

9. Use the keypad to enter a value. The default value is 0.01.

10. Press the **Enter** key to enter the threshold.

11. Press the **Escape** key repeatedly to return to the Main menu screen.

10.5 Shutoff Time

The YSI 556 MPS shuts off automatically after 30 minutes of inactivity. The shut off time may be changed as described below.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Shutoff time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Use the keypad to enter a value from 0 to 60 minutes. The default value is 30.

   NOTE: To disable the automatic shutoff feature, enter a zero (0).

4. Press the **Enter** key to enter the correct shutoff time.

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5. Press the **Escape** key repeatedly to return to the main menu screen.

10.6 Comma Radix

The user can toggle between a period (default) and comma for the radix mark by selecting this item and pressing the **Enter** key as follows:

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Comma radix** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press the **Enter** key. A check mark appears in the check box next to the comma radix selection indicating that the radix mark is a comma.

10.7 ID

This selection allows you to enter an identification name/number for your YSI 556 MPS. This ID name/number is logged in the header of each file.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **ID** selection. See Figure 10.1 Main Menu.

3. Use the keypad to enter an alphanumeric ID up to 15 characters in length. Refer to Section *2.9 Keypad Use*.

4. Press the **Enter** key to enter the ID.

5. Press the **Escape** key repeatedly to return to the main menu screen.

10.8 GLP Filename

This selection allows you to enter a different filename for the YSI 556 MPS Calibration Record file.

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NOTE: The default filename is the "556 PC board Serial Number.glp."

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **GLP Filename** selection. See Figure 10.1 Main Menu.
3. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.
4. Press the **Enter** key to enter the new filename.
5. Press the **Escape** key repeatedly to return to the main menu screen.

10.9 TDS Constant

This selection allows you to set the constant used to calculate Total Dissolved Solids (TDS). TDS in g/L is calculated by multiplying this constant times the specific conductance in mS/cm.

10.9.1 Changing the TDS Constant

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **TDS Constant** selection. See Figure 10.1 Main Menu.
3. Use the keypad to enter a value. Refer to Section *2.9 Keypad Use*. The default value is 0.65.
4. Press the **Enter** key to enter the correct TDS constant.
5. Press the **Escape** key repeatedly to return to the main menu screen.

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10.10 Barometer Units

The following information is only for instruments with the barometer option.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Barometer units** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press the **Enter** key. The Barometer units screen will appear.

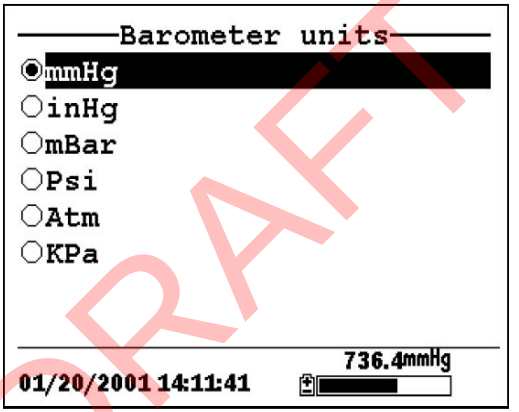

Figure 10.5 Data Filter Screen

A black dot indicates the currently selected units.

4. Use the arrow keys to highlight your desired barometric unit.

5. Press the **Enter** key to select your choice. A black dot will appear in the circle next to your selected units.

6. Press the **Escape** key repeatedly to return to the main menu screen.

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10.11 Calibrate Barometer

The optional barometer has been factory calibrated to provide accurate readings. However, some sensor drift may occur over time, requiring occasional calibration by the user, as follows:

1. Determine your local barometric pressure from an independent laboratory barometer or from your local weather service.

2. If the barometric pressure (BP) reading is from your local weather station, reverse the equation that corrects it to sea level.

   NOTE: For this equation to be accurate, the barometric pressure units must be in mmHg.

   True BP = (Corrected BP) – [2.5 * (Local Altitude/100)]

3. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

4. Use the arrow keys to highlight the **Calibrate barometer** selection on the system setup screen. See Figure 10.2 System Setup Screen.

5. Press the **Enter** key. The Calibrate Barometer screen is displayed.

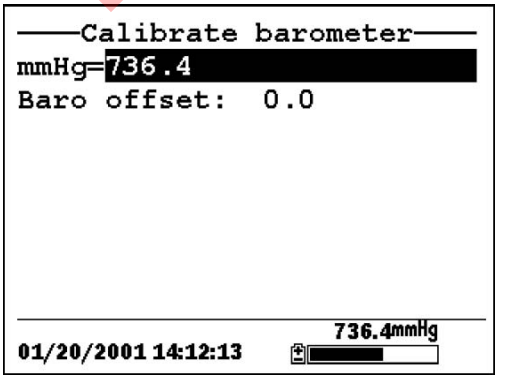

Figure 10.6 Barometer Calibration Screen

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6. Use the keypad to input the known barometric pressure value as determined in Step 2.

7. Press the **Enter** key. The new barometer reading is displayed as well as the approximate offset from the factory reading.

   NOTE: To return the sensor to the factory setting, subtract the offset amount from the current setting and repeat Steps 5 to 7.

8. Press the **Escape** key repeatedly to return to the main menu screen.

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11. Maintenance

11.1 Sensor Care and Maintenance

Once the sensors have been properly installed, remember that periodic cleaning and DO membrane changes are required.

11.1.1 DO Sensor

For best results, we recommend that the KCl solution and the membrane cap be changed at least once every 30 days.

1. It is important to recognize that oxygen dissolved in the sample is consumed during sensor operation. It is therefore essential that the sample be continuously stirred at the sensor tip. If stagnation occurs, your readings will be artificially low. Stirring may be accomplished by mechanically moving the sample around the sensor tip, or by rapidly moving the sensor through the sample. The rate of stirring should be at least 1 foot per second.

2. Membrane life depends on usage. Membranes will last a long time if installed properly and treated with care. Erratic readings are a result of loose, wrinkled, damaged, or fouled membranes, or from large (more than 1/8" diameter) bubbles in the electrolyte reservoir. If erratic readings or evidence of membrane damage occurs, you should replace the membrane and the electrolyte solution. The average replacement interval is two to four weeks.

3. If the membrane is coated with oxygen consuming (e.g. bacteria) or oxygen producing organisms (e.g. algae), erroneous readings may occur.

4. Chlorine, sulfur dioxide, nitric oxide, and nitrous oxide can affect readings by behaving like oxygen at the sensor. If you suspect erroneous readings, it may be necessary to determine if these gases are the cause.

5. Avoid any environment that contains substances that may attack the probe module and sensor materials. Some of these substances are concentrated acids, caustics, and strong solvents. The sensor materials that come in contact

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with the sample include FEP Teflon, acrylic plastic, EPR rubber, stainless steel, epoxy, polyetherimide and the PVC cable covering.

6. It is possible for the silver anode, which is the entire silver body of the sensor, to become contaminated. This will prevent successful calibration. To restore the anode, refer to Section *11.1.1 DO Sensor, Silver Anode Cleaning.*

7. For correct sensor operation, the gold cathode must always be bright. If it is tarnished (which can result from contact with certain gases), or plated with silver (which can result from extended use with a loose or wrinkled membrane), the gold surface must be restored. To restore the cathode, refer to Section *11.1.1 DO Sensor, Gold Cathode Cleaning.*

8. To keep the electrolyte from drying out, store the sensor in the transport/calibration cup with at least 1/8″ of water.

Silver Anode Cleaning

After extended use, a thick layer of AgCl builds up on the silver anode reducing the sensitivity of the sensor. The anode must be cleaned to remove this layer and restore proper performance. The cleaning can be chemical or mechanical:

Chemical Cleaning: Remove the membrane cap and soak the entire anode section in a 14% ammonium hydroxide solution for 2 to 3 minutes, followed by a thorough rinsing with distilled or deionized water. The anode should then be thoroughly wiped with a wet paper towel to remove the residual layer from the anode.

Mechanical Cleaning: Sand off the dark layer from the silver anode with 400 grit wet/dry sandpaper. Wrap the sandpaper around the anode and twist the sensor. Rinse the anode with clean water after sanding, followed by wiping thoroughly with a wet paper towel.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation***.**

Turn the instrument on and allow the system to stabilize for at least 30 minutes. If, after several hours, you are still unable to calibrate, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

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Gold Cathode Cleaning

For correct sensor operation, the gold cathode must be textured properly. It can become tarnished or plated with silver after extended use. The gold cathode can be cleaned by using the adhesive backed sanding disc and tool provided in the YSI 5238 Probe Reconditioning Kit.

Using the sanding paper provided in the YSI 5238 Probe Reconditioning Kit, wet sand the gold with a twisting motion about 3 times or until all silver deposits are removed and the gold appears to have a matte finish. Rinse the cathode with clean water after sanding, followed by wiping thoroughly with a wet paper towel. If the cathode remains tarnished, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation*.

11.1.2 DO Sensor Replacement

1. Remove the probe sensor guard.

⚠ **CAUTION:** Thoroughly dry the sensor so that no water enters the probe module sensor port when the sensor is removed.

2. Insert the long end of the hex key wrench into the small hole in the side of the probe module bulkhead. Turn the wrench counterclockwise and remove the screw. (You do not have to remove the screw all the way to release the sensor.)

3. Pull the old DO sensor module straight out of the probe module body.

   NOTE: The DO sensor is not threaded, it is keyed, so it cannot be removed by twisting.

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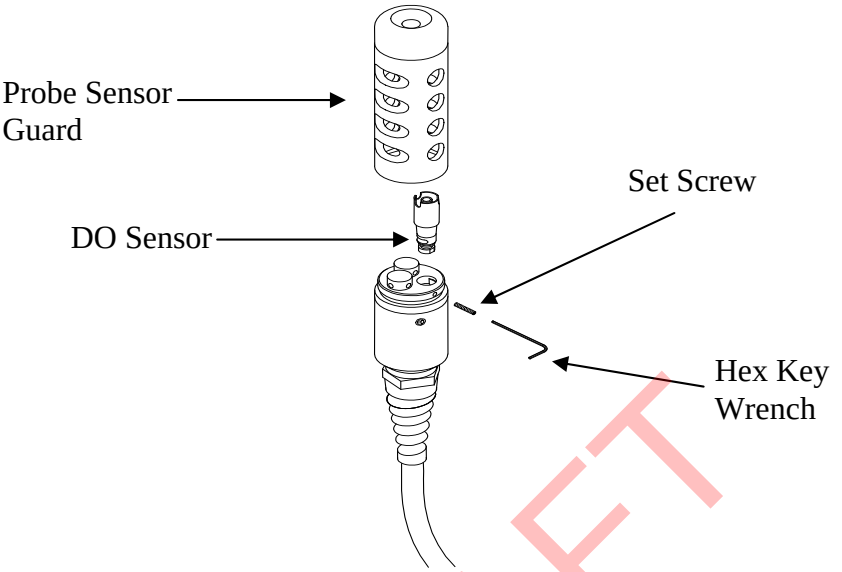

Figure 11.1 DO Sensor Replacement

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4. Insert the new DO sensor module. Make sure that the inside of the probe module sensor port and the o-ring on the sensor are clean, with no contaminants, such as grease, dirt, or hair. The DO sensor is keyed, or has a flat side, so that it cannot be aligned improperly.

   NOTE: Make sure the DO sensor bottoms out before the set screw is inserted.

5. Insert the set screw into the small hole in the side of the probe module bulkhead, and turn clockwise to rethread.

⚠ **CAUTION**: Make sure that you do not cross-thread the set screw. Use the hex key wrench to tighten the screw in properly, making sure that the screw does not stick out of the side of the probe module bulkhead. The probe sensor guard will not thread on properly and damage may result if the screw is allowed to stick out.

NOTE: The YSI 5563 DO sensor is shipped dry. A shipping membrane was installed to protect the electrode. A new membrane cap must be installed before the first use. Refer to Section 3.4.1 Sensor Installation.

11.1.3 YSI 5564 pH and 5565 Combination pH/ORP Sensor Cleaning

Cleaning is required whenever deposits or contaminants appear on the glass and/or platinum surfaces of these sensors or when the response of the sensor becomes slow.

1. Remove the sensor from the probe module.

2. Initially, simply use clean water and a soft clean cloth, lens cleaning tissue, or cotton swab to remove all foreign material from the glass bulb (YSI 5564 and YSI 5565) and platinum button (YSI 5565). Then use a moistened cotton swab to carefully remove any material that may be blocking the reference electrode junction of the sensor.

⚠ **CAUTION:** When using a cotton swab with the YSI 5564 or YSI 5565, be careful NOT to wedge the swab tip between the guard and the glass sensor. If necessary, remove cotton from the swab tip, so that the cotton can reach all parts of the sensor tip without stress.

NOTE: If good pH and/or ORP response is not restored by the above procedure, perform the following additional procedure:

1. Soak the sensor for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the cleaning solution.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water.

   NOTE: If good pH and/or ORP response is still not restored by the above procedure, perform the following additional procedure:

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1. Soak the sensor for 30-60 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most distributors. Be sure to follow the safety instructions included with the acid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the acid.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water. To be certain that all traces of the acid are removed from the sensor crevices, soak the sensor in clean water for about an hour with occasional stirring.

   NOTE: If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

1. Soak the sensor for approximately 1 hour in a 1 to 1 dilution of commercially available chlorine bleach.

2. Rinse the sensor with clean water and then soak for at least 1 hour in clean water with occasional stirring to remove residual bleach from the junction. (If possible, soak the sensor for period of time longer than 1 hour in order to be certain that all traces of chlorine bleach are removed.) Then re-rinse the sensor with clean water and retest.`

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11.1.4 Temperature/Conductivity Sensor Cleaning

The single most important requirement for accurate and reproducible results in conductivity measurement is a clean cell. A dirty cell will change the conductivity of a solution by contaminating it. The small cleaning brush included in the YSI 5511 Maintenance Kit is ideal for this purpose.

To clean the conductivity cell:

1. Dip the brush in clean water and insert it into each hole 1520 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

NOTE: In the event that deposits have formed on the electrodes, perform the following additional procedure:

1. Use a mild detergent solution in combination with the brush. Dip the brush in the solution and insert it into each hole 1520 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

NOTE: After cleaning, check the response and accuracy of the conductivity cell with a calibration standard.

NOTE: If this procedure is unsuccessful, or if sensor performance is impaired, it may be necessary to return the sensor to a YSI authorized service center for service, Refer to *Appendix E Customer Service*.

The temperature portion of the sensor requires no maintenance.

11.2 Upgrading YSI 556 MPS Software

1. Access the YSI Environmental Software Downloads page as described in *Appendix G EcoWatch* Step 1 through 3.

2. Click on the **YSI Instruments Software Updates** link (or scroll down until you see YSI 556 MPS).

3. Click on the file icon to the right of the **YSI 556 MPS** listing and save the file to a temporary directory on your computer.

4. After the download is complete, run the file (that you just downloaded) and follow the on screen instructions to install the YSI Code Updater on your computer. If you encounter difficulties, contact YSI customer service for advice. Refer to *Appendix E Customer Service*.

5. If necessary, disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

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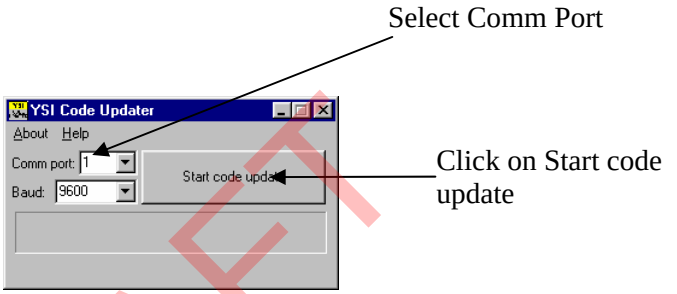
6. Connect the YSI 556 MPS to a serial port of your computer via the 655173 PC interface cable. See Figure 8.6 Computer/Instrument Interface.

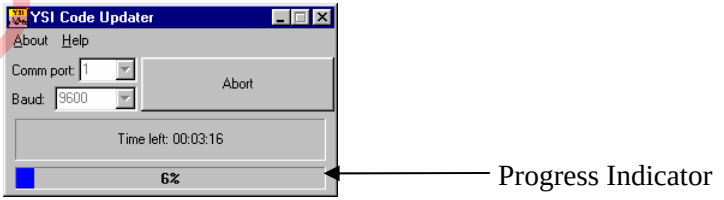
7. Press the **On/off** key on the YSI 556 MPS to display the run screen.

8. Run the YSI Code Updater software that you just installed on your computer. The following window will be displayed:



9. Set the Comm port number to match the port that you connected the 655173 PC Interface Cable to, then click on the Start Code Update button.

The YSI 556 MPS screen will blank out and a progress indicator will be displayed on the PC.



When the update is finished (indicated on the PC screen), the YSI 556 MPS will return to the Run screen. See Figure 7.1 Run Screen.

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10. Close the YSI Code Updater window (on the PC) by clicking on the "X" in the upper right corner of the window.

11. Disconnect the YSI 556 MPS from the 655173 PC interface cable and reconnect it to the YSI 5563 Probe Module. Refer to Section *3.6 Instrument/Cable Connection*.

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12. Storage

Proper storage between periods of usage will not only extend the life of the sensors, but will also ensure that the unit will be ready to use as quickly as possible in your next application.

12.1 General Recommendations for Short Term Storage

No matter what sensors are installed in the instrument, it is important to keep them moist without actually immersing them in liquid. Immersing them could cause some of them to drift or result in a shorter lifetime.

YSI recommends that short term storage of all multi-parameter instruments be done by placing approximately 1/2 inch of tap water in the transport/calibration cup that was supplied with the instrument, and by placing the probe module with all of the sensors installed into the cup. The use of a moist sponge instead of a 1/2 inch of tap water is also acceptable, as long as its presence does not compromise the attachment of the cup to the probe module. The transport/calibration cup should be sealed to prevent evaporation.

NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

⚠ **CAUTION**: The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

NOTE: If the storage water (tap water) is accidentally lost during field use, environmental water can be used.

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12.2 General Recommendations for Long Term Storage

12.2.1 Probe Module Storage

1. Remove the pH or pH/ORP sensor from the probe module and store according to the individual sensor storage instructions found in Section *12.2.2 Sensor Storage*.

2. Seal the empty port with the provided port plug.

   NOTE: Leave the conductivity/temperature sensor and

dissolved oxygen sensor, with membrane cap still on, in the probe module.

3. Place 1/2″ of water, deionized, distilled or tap, in the transport/calibration cup.

⚠ **CAUTION:** The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

4. Insert the probe module into the cup.

NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

12.2.2 Sensor Storage

Temperature/Conductivity Sensor

No special precautions are required. Sensor can be stored dry or wet, as long as solutions in contact with the thermistor and conductivity electrodes are not corrosive (for example, chlorine bleach). However, it is recommended that the sensor be cleaned with the provided brush prior to long term storage. Refer to Section *11.1.4 Temperature/Conductivity Sensor Cleaning.*

pH and Combination pH/ORP Sensor

The key to sensor storage is to make certain that the reference electrode junction does not dry out. Junctions which have been allowed to dry out due to improper storage procedures can usually be rehydrated by soaking the sensor for several hours (overnight is recommended) in a solution which is 2 molar in potassium chloride. If potassium chloride solution is not available, soaking the sensor in tap water or commercial pH buffers may restore sensor function. However in some cases the sensor may have been irreparably damaged by the dehydration and will require replacement.

⚠ **CAUTION:** Do not store the sensor in distilled or deionized water as the glass sensor may be damaged by exposure to this medium.

1. Remove the pH or pH/ORP sensor from the probe module.

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2. Seal the empty port with the provided port plug.

3. Place the sensor in the storage vessel (plastic boot or bottle) which was on the sensor at delivery. The vessel should contain a solution which is 2 molar in potassium chloride.

   NOTE: Make certain that the vessel is sealed to prevent evaporation of the storage solution.

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13. Troubleshooting

The following sections describe problems you may encounter when using the YSI 556 MPS and provides suggestions to overcome the symptom.

PROBLEM	POSSIBLE SOLUTION
Display Problems	
No display is visible after pressing the on/off key.	If C cells are used, make certain that they are installed properly with regard to polarity and that good batteries are used. If a rechargeable battery pack is used, place the pack in the instrument and charge for 30 minutes.
Instrument software appears to be locked up as evidenced by no response to keypad entries or display not changing.	First, attempt to reset the instrument by simply turning off and then on again. If this fails, remove battery power from the instrument for 30 seconds and then reapply power. When using C cells, remove the battery lid and one of the batteries; when using the rechargeable battery pack, remove the pack completely from the instrument. After 30 seconds replace the battery or battery pack and check for instrument function.
The 556 display flashes and the instrument speaker makes a continuous clicking sound.	The battery voltage is low. Change to new C cells or recharge the 6117 battery pack.
Water Damage to Instrument	
Leakage detected in battery compartment when using C cells.	Dispose of batteries properly. Dry the battery compartment using compressed air if possible. If corrosion is present on battery terminals, contact YSI Customer Service.
Water has contacted rechargeable battery pack.	Remove battery pack immediately. Send battery pack to YSI Product Service for evaluation. **CAUTION**: DO NOT REUSE BATTERY PACK UNTIL YSI PRODUCT SERVICE HAS EVALUATED IT.
Leakage suspected into the main cavity of the instrument case.	Remove the batteries immediately. Return the instrument to YSI Product Service.

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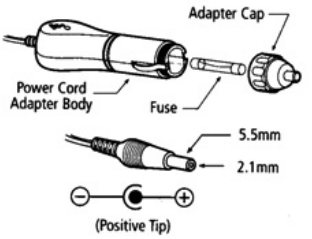
PROBLEM	POSSIBLE SOLUTIONS
Optional Cigarette Lighten Charger	
Power cord fuse blown.  	1. Unscrew adapter's cap, remove tip and pull out fuse. 2. Replace fuse with a new 2-amp fast-blow fuse from an electronics store such as Radio Shack. 3. Reassemble the adapter and securely screw the cap back onto the adapter body.
File Problems	
Upload of files from YSI 556 MPS to PC fails	1. Make sure that cable is connected properly to both 556 and PC. 2. Make certain that the proper Comm port is selected in EcoWatch for Windows.
Barometer data is not stored with sensor data file.	Make sure **Store barometer** is active in the 556 **Logging setup** menu.
Site Descriptions in the **Site List** are "grayed-out" and not available for appending files with additional data.	There is a parameter mismatch between the current 556 setup and that initially used. Change the current logging and sensor setup to match the setup that was initially used to create the file.
Sensor Problems	
Dissolved Oxygen reading is unstable or inaccurate. Out of Range message appears during calibration.	Sensor not properly calibrated. Follow DO cal procedures.
	Membrane not properly installed or may be punctured. Replace membrane cap.
	DO sensor electrodes require cleaning. Follow DO cleaning procedure. Use 5511 Maintenance kit.
	Water in sensor connector. Dry connector; reinstall sensor.
	Algae or other contaminant clinging to DO sensor. Rinse DO sensor with clean water.
	Barometric pressure entry is incorrect. Repeat DO cal procedure.
	Calibrated at extreme temperature. Recalibrate at (or near) sample temperature.
	DO sensor has been damaged. Replace sensor.
	Internal failure. Return probe module for service.

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PROBLEM	POSSIBLE SOLUTIONS
Sensor Problems	
pH or ORP readings are unstable or inaccurate. Out of Range message appears during calibration.	Sensor requires cleaning. Follow sensor cleaning procedure.
	Sensor requires calibration. Follow cal procedures.
	pH sensor reference junction has dried out from improper storage. Soak sensor in tap water or buffer 4 until readings become stable.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace sensor.
	Calibration solutions out of spec or contaminated with other solution. Use new calibration solutions
	ORP fails Zobell check. Take into account temperature dependence of Zobell solution readings.
	Internal failure. Return probe module for service.
Conductivity unstable or inaccurate. Out of Range message appears during calibration	Conductivity improperly calibrated. Follow calibration procedure.
	Conductivity sensor requires cleaning. Follow cleaning procedure.
	Conductivity sensor damaged. Replace sensor.
	Calibration solution out of spec or contaminated. Use new calibration solution.
	Internal failure. Return probe module for service.
	Calibration solution or sample does not cover entire sensor. Immerse sensor fully.
Temperature, unstable or inaccurate	Water in connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace the 5560 sensor.
Installed sensor has no reading	The sensor has been disabled. Enable sensor.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace sensor.
	Report output improperly set up. Set up report output.
	Internal failure. Return probe module for service.

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If these guidelines and tips fail to correct your problem or if any other symptoms occur, contact YSI Customer Service for Advice. Refer to *Appendix E Customer Service*.

14. Appendix A YSI 556 MPS Specifications

For the most recent product specifications, please visit the YSI website: www.ysi.com

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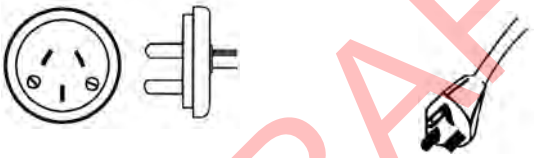
15. Appendix B Instrument Accessories

ITEM #	ACCESSORY
5563-4	4m Cable with DO/temp/conductivity
5563-10	10m Cable with DO/temp/conductivity
5563-20	20m Cable with DO/temp/conductivity
5564	pH Kit
5565	pH/ORP Kit
6118	Rechargeable Battery Pack Kit for use in US
5094	Rechargeable Battery Pack Kit with universal charger and three adapter cables for use in international applications 
5095	Rechargeable Battery Pack Kit with universal charger and two adapter cables for use in international applications 
5083	Flow Cell – probe module is secured in the flow cell and groundwater is pumped through it. Displaced volume approx. 475 ml
3059	Flow Cell, low volume. Displaced volume approx. 200 ml
116505	Battery Lid
616	Charger, Cigarette Lighter – used to power up the instrument from a car's cigarette lighter
4654	Tripod
614	Ultra Clamp, C Clamp –used to clamp the instrument to a table top or car dashboard
6081	Large Carrying Case, Hard-sided
5085	Hands-free Harness
5065	Carrying Case, Form-fitted, for use in the field – has a clear vinyl window, shoulder strap, belt loop strap and hand strap

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16. Appendix C Required Federal Communications Notice

The Federal Communications Commission defines this product as a computing device and requires the following notice.

This equipment generates and uses radio frequency energy and if not installed and used properly, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class A or Class B computing device in accordance with the specification in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna
- Relocate the computer with respect to the receiver
- Move the computer away from the receiver
- Plug the computer into a different outlet so that the computer and receiver are on different branch circuits.

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet, prepared by the Federal Communications Commission, helpful: "How to Identify and Resolve Radio-TV Interference Problems". This booklet is available from the U.S. Government Printing Office, Washington, D.C. 20402, Stock No.0004-000-00345-4.

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17. Appendix D Health Safety

YSI Conductivity Solutions: 3161, 3163, 3165, 3167, 3168, 3169

INGREDIENTS:

- Iodine
- Potassium Chloride
- Water

WARNING: INHALATION MAY BE FATAL

⚠ ***CAUTION:*** **AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY EVOLVE TOXIC FUMES IN FIRE.**

Harmful if ingested or inhaled. Skin or eye contact may cause irritation. Has a corrosive effect on the gastro-intestinal tract, causing abdominal pain, vomiting, and diarrhea. Hyper-sensitivity may cause conjunctivitis, bronchitis, skin rashes etc. Evidence of reproductive effects.

FIRST AID:
INHALATION: Remove victim from exposure area. Keep warm and rest. In severe cases seek medical attention.
SKIN CONTACT: Remove contaminated cloth immediately. Wash affected area thoroughly with large amounts of water. In severe cases seek medical attention.
EYE CONTACT: Wash eyes immediately with large amounts of water, (approx. 10 minutes). Seek medical attention immediately.
INGESTION: Wash out mouth thoroughly with large amounts of water. Seek medical attention immediately.

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YSI pH 4.00, 7.00, y 10.00: 3821, 3822, 3823

pH 4 INGREDIENTS:

- Potassium Hydrogen Phthalate
- Formaldehyde
- Water

pH 7 INGREDIENTS:

- Sodium Phosphate, Dibasic
- Potassium Phosphate, Monobasic
- Water

pH 10 INGREDIENTS:

- Potassium Borate, Tetra
- Potassium Carbonate
- Potassium Hydroxide
- Sodium (di) Ethylenediamine Tetraacetate
- Water

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⚠ **CAUTION -AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

Inhalation may cause severe irritation and be harmful. Skin contact may cause irritation; prolonged or repeated exposure may cause Dermatitis. Eye contact may cause irritation or conjunctivitis. Ingestion may cause nausea, vomiting and diarrhea.

FIRST AID:
INHALATION – Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.
SKIN CONTACT – Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.
EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.

INGESTION – If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

YSI Zobell Solution: 3682

INGREDIENTS:

- Potassium Chloride
- Potassium Ferrocyanide Trihydrate
- Potassium Ferricyanide

⚠ **CAUTION -AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

May be harmful by inhalation, ingestion, or skin absorption. Causes eye and skin irritation. Material is irritating to mucous membranes and upper respiratory tract. The chemical, physical, and toxicological properties have not been thoroughly investigated.

Ingestion of large quantities can cause weakness, gastrointestinal irritation and circulatory disturbances.

FIRST AID:
INHALATION – Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.
SKIN CONTACT – Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.
EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.
INGESTION – If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

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18. Appendix E Customer Service

18.1 Ordering and Technical Support

Telephone:	800 897 4151 (US)
	+1 937 767 7241 (Globally)
	Monday through Friday, 8:00 AM to 5:00 ET
Fax:	+1 937 767 9353 (orders)
	+1 937 767 1058 (technical support)
Email:	environmental@ysi.com or proseries@ysi.com
Mail:	YSI Incorporated
	1725 Brannum Lane
	Yellow Springs, OH 45387 USA
Website:	www.ysi.com

18.2 YSI Authorized Service Centers

YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit www.ysi.com and click ‘Support’ or contact YSI Technical Support directly at 800-897-4151.

When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for a YSI Service Center to accept the instrument for service. The form may be downloaded from www.ysi.com by clicking on the ‘Support” tab, then the Product Return Form button.

18.3 Cleaning Instructions

Equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected before being serviced. Biological contamination is presumed for any instrument, probe, or other device that has been used with body fluids or tissues, or with wastewater. Radioactive contamination is presumed for any instrument, probe or other device that has been used near any radioactive source.

If an instrument, probe, or other part is returned or presented for service without a Cleaning Certificate, and if in our opinion it represents a potential

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biological or radioactive hazard, our service personnel reserve the right to withhold service until appropriate cleaning, decontamination, and certification has been completed. We will contact the sender for instructions as to the disposition of the equipment. Disposition costs will be the responsibility of the sender.

When service is required, either at the user's facility or at a YSI Service Center, the following steps must be taken to ensure the safety of service personnel.

- In a manner appropriate to each device, decontaminate all exposed surfaces, including any containers. 70% isopropyl alcohol or a solution of 1/4-cup bleach to 1-gallon tap water is suitable for most disinfecting. Instruments used with wastewater may be disinfected with .5% Lysol if this is more convenient to the user.
- The user shall take normal precautions to prevent radioactive contamination and must use appropriate decontamination procedures should exposure occur.
- If exposure has occurred, the customer must certify that decontamination has been accomplished and that no radioactivity is detectable by survey equipment.
- Any product being returned to the YSI Repair Center should be packed securely to prevent damage.
- Cleaning must be completed and certified on any product before returning it to YSI.

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18.4 Packing Procedure

- Clean and decontaminate items to ensure the safety of the handler.
- Complete and include the Cleaning Certificate.
- Place the product in a plastic bag to keep out dirt and packing material.
- Use a large carton, preferably the original, and surround the product completely with packing material.
- Insure for the replacement value of the product.

18.5 Warranty

The instrument is warranted for three years against defects in workmanship and materials when used for its intended purposes and maintained according to instructions. The probe module and cables are warranted for one year. The dissolved oxygen, temperature/conductivity, pH, and pH/ORP combination sensors are warranted for one year. Damage due to accidents, misuse, tampering, or failure to perform prescribed maintenance is not covered. The warranty period for chemicals and reagents is determined by the expiration date printed on their labels. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

Limitation of Warranty

This Warranty does not apply to any YSI product damage or failure caused by (i) failure to install, operate or use the product in accordance with YSI's written instructions, (ii) abuse or misuse of the product, (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure, (iv) any improper repairs to the product, (v) use by you of defective or improper components or parts in servicing or repairing the product, or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI'S LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

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19. Appendix F Ferrite Bead Installation

⚠ **WARNING:** If you are using your YSI 556 in a European Community (CE) country or in Australia or New Zealand, you must attach a ferrite bead to the 655173 PC Interface Cable and the YSI 6117 Charger Adapter Cable in order to comply with the Residential, Commercial and Light Industrial Class B Limits for radio-frequency emissions specified in EN55011 (CISPR11) for Industrial, Scientific and Medical laboratory equipment. These ferrite assemblies are supplied as part of cable kits.

1. Make a small loop (approximately 5 cm in diameter) in the cable near the YSI 556 MS-19 connector.

2. Lay the open ferrite bead assembly under the loop with the cable cross-over position within the cylinder of the ferrite bead.

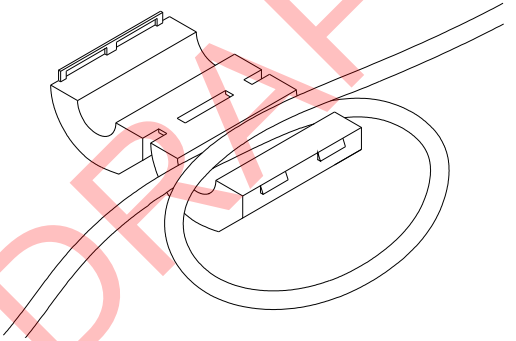

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Figure 19.1 Ferrite Bead Installation

3. Snap the two pieces of the bead together making certain that the tabs lock securely.

4. When the installation is complete, the 655173 and 6117 cables should resemble the following drawings.

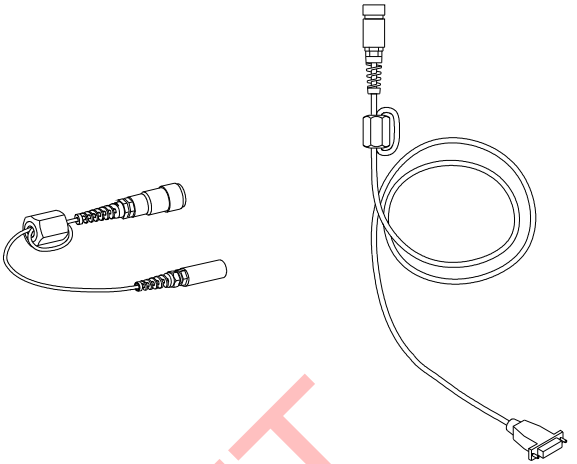

Figure 19.2 Cables with Ferrite Beads

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20. Appendix G EcoWatch

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. EcoWatch is a powerful tool that can also be used with YSI 6-series sondes. Many features of the software will only be utilized by advanced users or are not relevant to the 556 MPS at all. This section is designed in tutorial format to familiarize you with the commonly used features of EcoWatch so that it will be possible to:

- Upload data from a 556 MPS to a PC
- Assemble plots and reports of your data
- Zoom in on certain segments of the plots of your data to facilitate analysis
- Show statistical data for your studies
- Export data in spreadsheet-compatible formats
- Print plots and reports

The advanced features of EcoWatch can be explored by downloading a 6-series manual from the YSI Web Site (www.ysi.com), purchasing a hard copy of the manual through YSI Customer Service (Item # 069300), or utilizing the on-line help feature of the software.

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20.1 Installing EcoWatch for Windows

EcoWatch for Windows is available at no cost via a download from the YSI Web Site – www.ysi.com

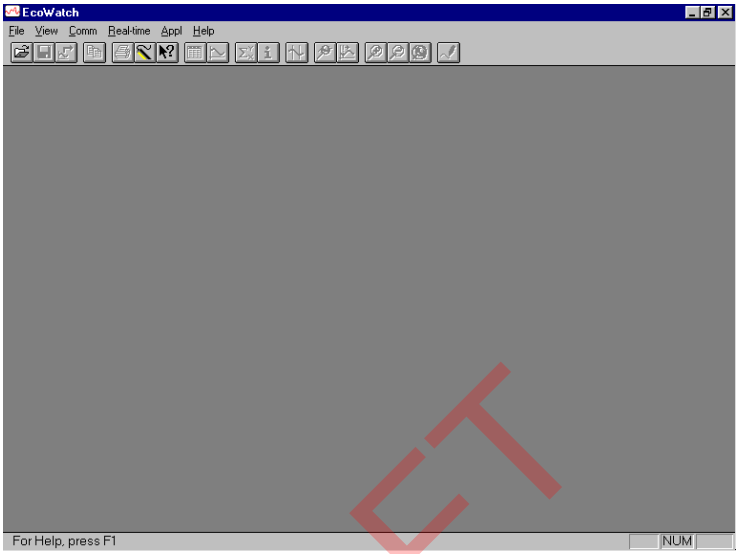
20.2 EcoWatch Tutorial

This EcoWatch tutorial is designed to teach you the commonly used operations associated with the software when used with your 556 MPS.

After you have uploaded a file, Refer to Section 8.4 Upload to PC, you will see two files in the C:\ECOWWIN\DATA directory; the file you transferred and a file supplied by YSI designated SAMPLE.DAT. This SAMPLE.DAT file is referred to in the remainder of this tutorial section. After following the instructions below for the analysis of SAMPLE.DAT, you apply the same analysis to the data file which was uploaded from your 556 MPS to assure that you are familiar with the basic features and capabilities of EcoWatch for Windows.

To start the analysis of the SAMPLE.DAT file, note that a shortened menu

bar is visible and many of the tools in the toolbar appear dimmed or “grayed out” before any file is opened (see below).



Full activation of EcoWatch features will be realized after a file is opened.

To open the sample data file:

1. Click the **File** menu  button in the toolbar.
2. Select the **SAMPLE.DAT** file.
3. Click **OK** to open the file.

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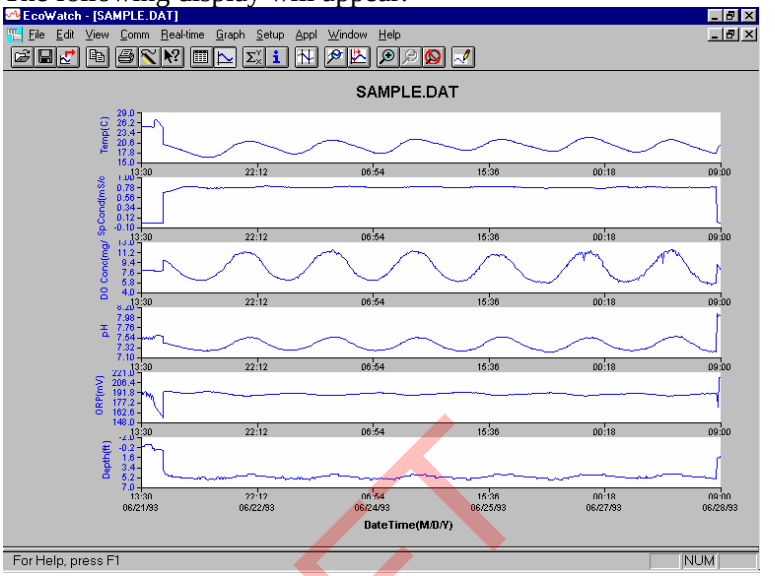
The following display will appear:



Note that the data in this file appears as a graph of temperature, specific conductance, dissolved oxygen, pH, ORP, and depth, all versus time. The graphs are scaled automatically so that all data fits comfortably on the computer screen. Note also that this data file was obtained with a 6-series sonde for which a depth sensor is available. Depth is NOT a current parameter for the 556 MPS.

The **Table** and **Graph** buttons on the toolbar are on/off switches that are used to display or hide the graph and table pages respectively. When displaying a graph and a table at the same time, you can control the relative size of the two pages by placing the cursor over the small bar that separates them and

then dragging it to the desired location. Click the **Table** button to generate the following dual display of data.

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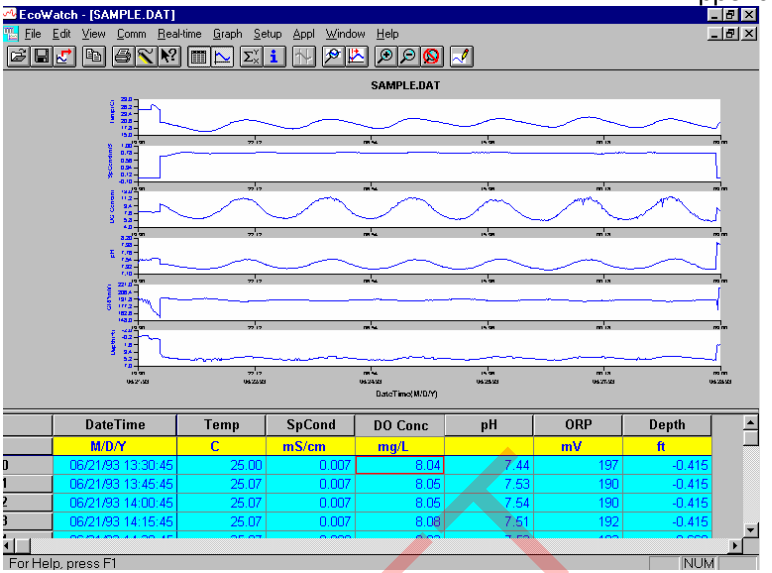
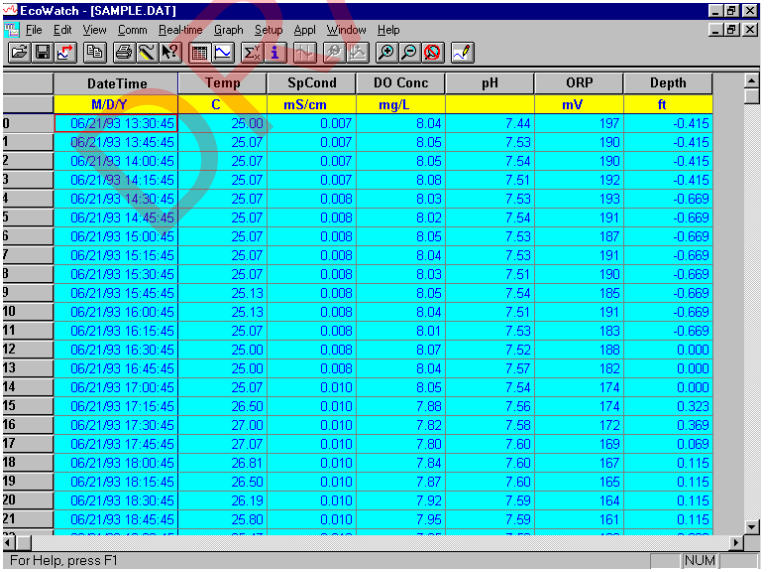


	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415

Now click the **Graph** button (turn it off) to display only a report of your data as shown below. Note that the size of the report can be varied by clicking on the and buttons in the Toolbar.



	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415
4	06/21/93 14:30:45	25.07	0.008	8.03	7.53	193	-0.669
5	06/21/93 14:45:45	25.07	0.008	8.02	7.54	191	-0.669
6	06/21/93 15:00:45	25.07	0.008	8.05	7.53	187	-0.669
7	06/21/93 15:15:45	25.07	0.008	8.04	7.53	191	-0.669
8	06/21/93 15:30:45	25.07	0.008	8.03	7.51	190	-0.669
9	06/21/93 15:45:45	25.13	0.008	8.05	7.54	185	-0.669
10	06/21/93 16:00:45	25.13	0.008	8.04	7.51	191	-0.669
11	06/21/93 16:15:45	25.07	0.008	8.01	7.53	183	-0.669
12	06/21/93 16:30:45	25.00	0.008	8.07	7.52	188	0.000
13	06/21/93 16:45:45	25.00	0.008	8.04	7.57	182	0.000
14	06/21/93 17:00:45	25.07	0.010	8.05	7.54	174	0.000
15	06/21/93 17:15:45	26.50	0.010	7.88	7.56	174	0.323
16	06/21/93 17:30:45	27.00	0.010	7.82	7.58	172	0.369
17	06/21/93 17:45:45	27.07	0.010	7.80	7.60	169	0.069
18	06/21/93 18:00:45	26.81	0.010	7.84	7.60	167	0.115
19	06/21/93 18:15:45	26.50	0.010	7.87	7.60	165	0.115
20	06/21/93 18:30:45	26.19	0.010	7.92	7.59	164	0.115
21	06/21/93 18:45:45	25.80	0.010	7.95	7.59	161	0.115

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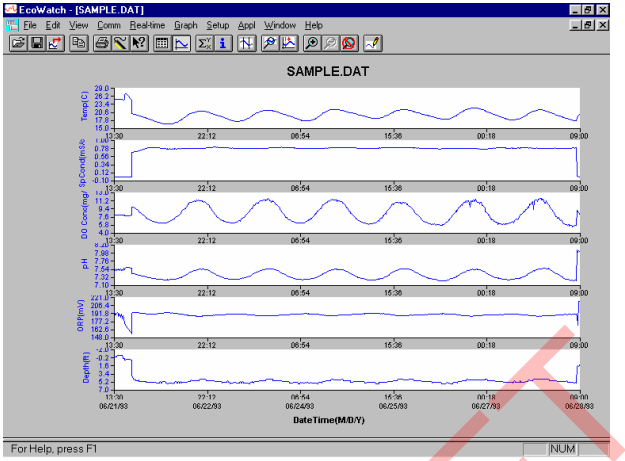
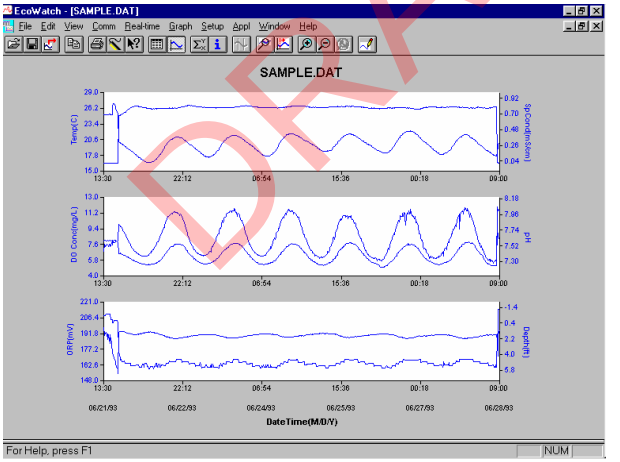
Now return to the original graphic display by toggling the **Table** button "off" and **Graph** button "on".



From the **Setup** menu, click **Graph**. Click **2 Traces per Graph** and notice that the parameters are now graphed in pairs for easy comparison of parameters.



Click **1 Trace per Graph** to return the display to the original setting. Move the cursor to any position in the graph, then click and hold the right mouse button.

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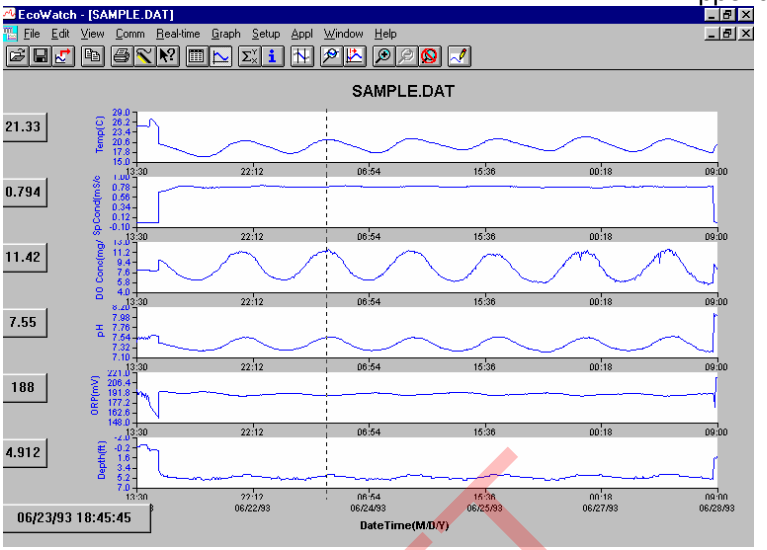

Note that the exact measurements for this point in time are displayed to the left of the graph. While holding down the right mouse button, move to another area on the graph. Notice how the measurements change as you move. When you release the mouse button, the display returns to normal.

To view statistical information for the study, click the **Statistics** button on the toolbar. On the statistics window, click on any min or max value to display the time when it occurred.

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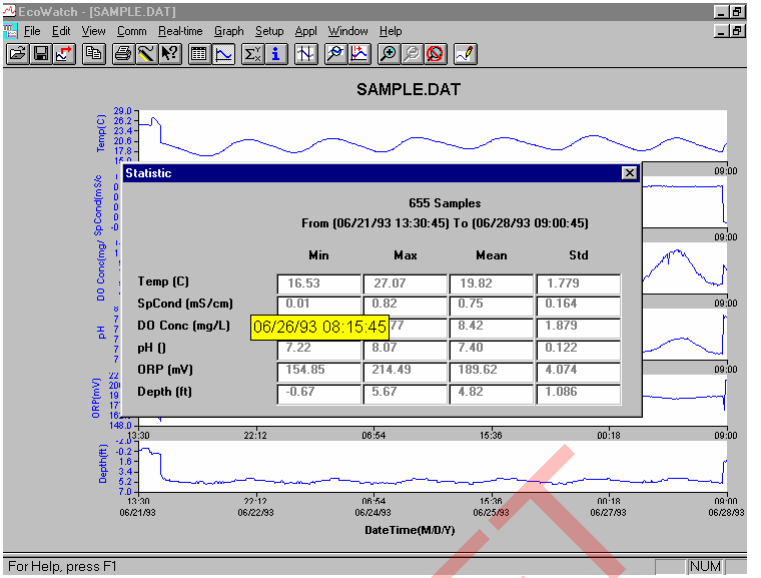
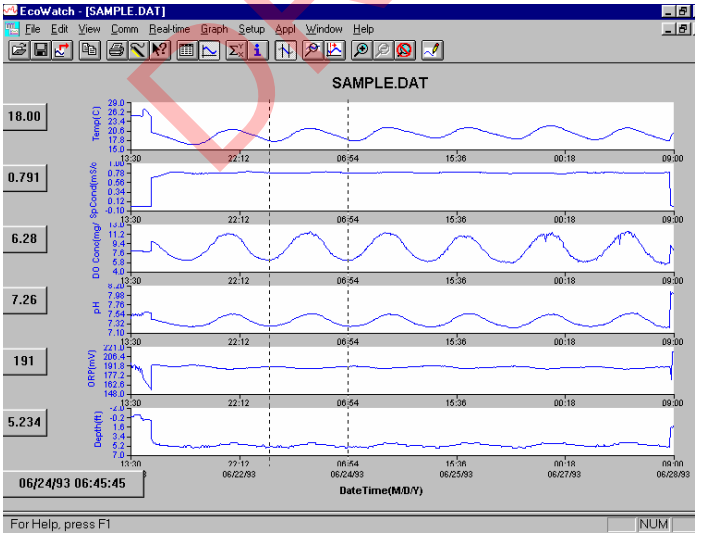


After viewing statistics, click the "x" at the upper right to close the window and return to the normal display.

Now click on the delimiter icon in the toolbar and then move the displayed icon to the graph. Click at the two points shown by dotted lines in the display below, being sure that the first click is to the left of the second.



The data between the two selected points will then be graphed in higher resolution as shown below.

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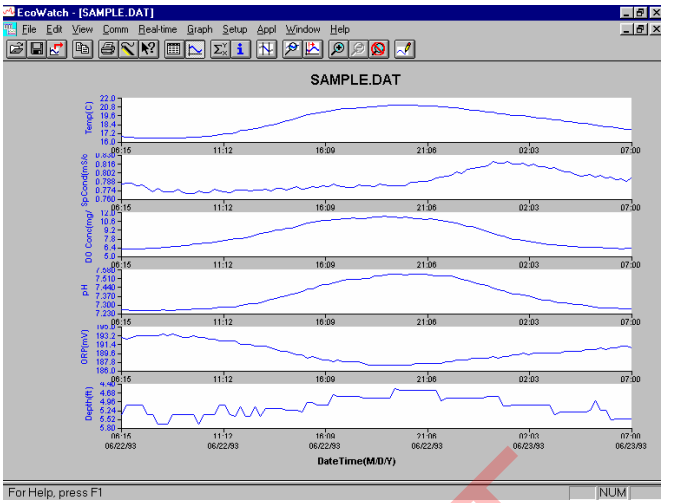
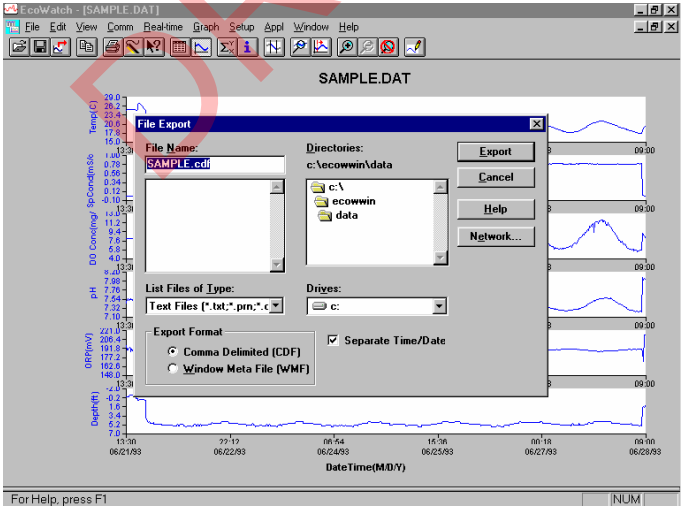


To return to the complete data set, select **Graph** from the toolbar and then click **Cancel Limits**.

Now select the  icon from the Toolbar to create a new data file which will allow your data to be imported into spreadsheets. Select the default export settings for a Comma Delimited File (.CDF) and click OK. A new spreadsheet-importable file (SAMPLE.CDF) is now present in the same folder as the SAMPLE.DAT file.



Now select the  icon from the toolbar to print the plot. Accept the default settings and click OK to complete the printing operation.

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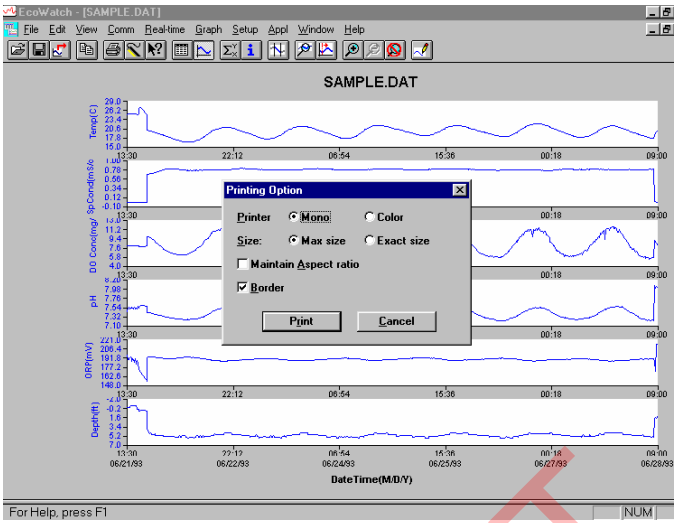
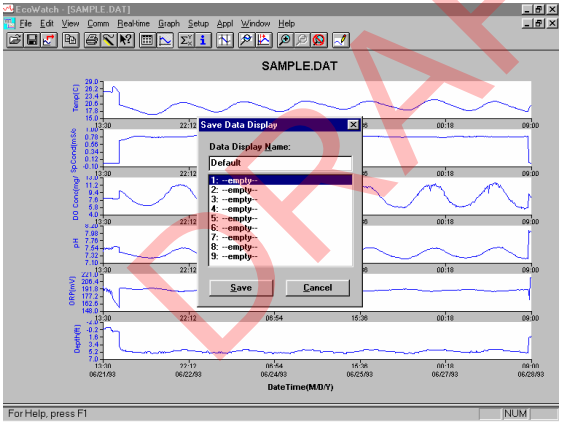


Finally, end the tutorial by saving the **Data Display** in the format shown. From the File menu, click **Save Data Display**.



Then type “Default” for the file name and click **Save**. The parameters, colors, format, and x-axis time interval associated with the current display are now saved and can be accessed any time in the future. Nine different data displays may be saved for any data file. You can easily switch between various displays of the data. The data files can be accessed by clicking **Load Data Display** from the file menu and then selecting the desired presentation.

20.2.1 Summary of Toolbar Capability

The EcoWatch toolbar includes buttons for some of the most common commands in EcoWatch, such as **File Open**. To display or hide the toolbar,

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open the **View** menu and click on the **Toolbar** command. A check mark appears next to the menu item when the toolbar is displayed.

The toolbar is displayed across the top of the application window, below the menu bar.

Click to:

 Open an existing data file (.DAT). EcoWatch displays the **Open** dialog box, in which you can locate and open the desired file.

 Save the working Data Display of the active data file. EcoWatch displays the **Save Data Display** dialog box in which you can overwrite existing Data Display or save to a new one.

 Export data as a graph in Window Meta File (.WMF) format or as data in Comma Delimited (.CDF) format.

 Copy the whole graph page or data from the selection on the table to the clipboard.

 Print the active graph page or table page depending on which one is currently active.

 Open a new terminal window to communicate with the sonde.

 Access context sensitive help (Shift+F1).

 Toggle table window during file processing.

 Toggle graph window during file processing.

 Display study statistics.

 Display study info.

 Limit the data to be processed.

 Enlarge a selective portion of graph.

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 Center the graph under the cursor.

 Enlarge graph of table 20%.

 Reduce graph of table 20%.

 Return graph or table to its normal size (unzoom).

 Redraw the graph.

20.2.2 Other capabilities

The above tutorial and function list for the toolbar provide basic information to allow you to view and analyze the field data which was stored in your 556 MPS. Some of the other commonly used capabilities of EcoWatch which the user may want to explore are listed below:

- Customize the units for each parameter, e.g., report uS/cm instead of mS/cm for conductivity.
- Customize the order of parameters in each plot or report.
- Customize the colors and fonts of each data display.
- Manually scale the y-axis sensitivity for each parameter.
- Merging of two or more data files with compatible parameter formats
- View information about the study such as number of points, instrument serial number, etc. which was stored in the 556 with the data.
- Print data reports in different statistical formats.
- Create plots of parameter vs. parameter rather than parameter vs. time.

These additional features of EcoWatch for Windows are explained in detail in the YSI 6-series manual (which can be downloaded at no cost from the YSI Web Site as described above) and the Help selection in the EcoWatch menu bar. To purchase a hard copy of the 6-series manual, contact YSI Customer Service using the contact information in *Appendix E Customer Service*.

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21. Appendix H Calibration Record Information

When your YSI 556 MPS sensors are initially calibrated, relevant information about the sensors will be stored in a separate file in the YSI 556 MPS memory.

NOTE: This file, by default, will have the name "556 Circuit Board Serial Number.glp." The circuit board serial number is assigned at the factory and has a hexadecimal format such as 000080A4. Thus the default calibration record file would be designated 00080A4.glp. Refer to Section *10.7 GLP Filename* to change the filename.

The information in the calibration record will track the sensor performance of your instrument and should be particularly useful for programs operating under Good Laboratory Practices (GLP) protocols.

21.1 Viewing the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

Follow the procedures outlined in Section *8.3 View File*.

21.2 Uploading the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

Follow the procedures outlined in Section *8.4 Upload to PC*.

21.3 Understanding the Calibration Record (.glp) File

1. Open a calibration record file. Refer to Section 8.3 View File.
2. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

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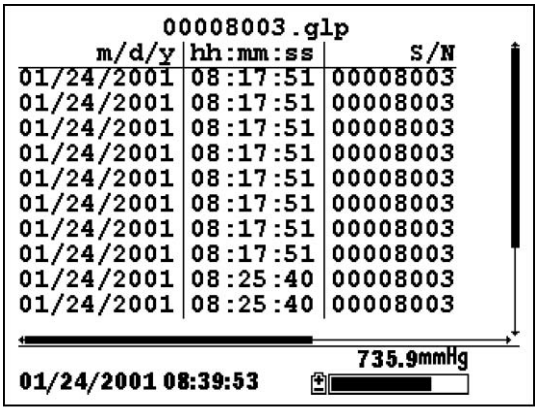

Figure 21.1Calibration Record Screen 1

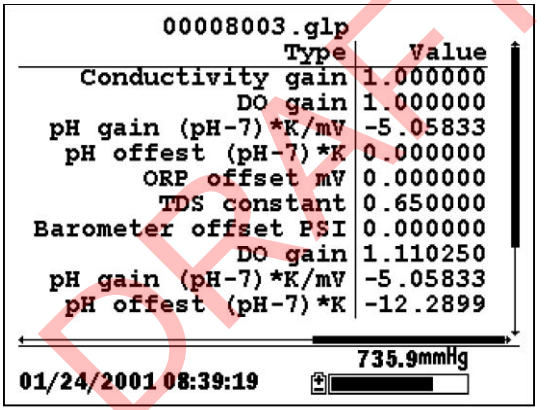

Figure 21.2 Calibration Record Screen 2

NOTE: Each sensor (not parameter) is characterized by either 1 line (Conductivity, Dissolved Oxygen, ORP, TDS, or Barometer (Optional)) or 2 lines (pH) of calibration documentation.

The left hand portion of each calibration entry shows the date and time that a calibration of a particular sensor was performed. In addition, each calibration entry is characterized by the instrument serial number, as defined by YSI. See Figure 21.1 Calibration Record Screen 1. The right hand portion shows the YSI designation of the calibration constants and their values after their calibration has been performed. A more detailed description of the calibration constants is provided below:

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- **Conductivity Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the calculated cell constant divided by the typical value of the cell constant ($5\ cm^{-1}$).
- **DO Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the sensor current at the time of calibration divided by the typical value of the sensor current (15 uA).
- **pH Gain** – A number which basically represents the sensitivity of the pH sensor. To remove the effect of temperature on the slope of the relationship of probe output in mv versus pH, the value of pH/mv is multiplied by the temperature in degrees Kelvin (K).
- **pH Offset** – A number which basically represents the offset (or intercept) of the relationship of probe output in mv versus pH, the value of pH is multiplied by the temperature in degrees Kelvin (K).

Anytime you perform a calibration, information concerning the calibration constants will be logged to the Calibration Record file (.glp file). However, if the **Delete All Files** command is used, Refer to Section *8.6 Delete All Files*, the Calibration Record file will also be lost. It is critical that this file should be uploaded to your PC prior to issuing a **Delete All Files** command. Refer to Section *8.4 Upload to PC*.

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YSI Environmental
1700/1725 Brannum Lane
Yellow Springs, OH
45387 USA
937.767.7241
937.767.9353 fax
environmental@YSI.com
www.YSI.com

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Item # 655279
Rev D
Drawing # A655279
August 2009
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MiniRAE 3000
User's Guide

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RAE SYSTEMS by Honeywell

Rev. F
February 2016
P/N 059-4020-000

FCC Information

Contains FCC ID: PI4411B or SU3RM900

The enclosed device complies with part 15 of the FCC rules. Operation is subject to the following conditions: (1) This device may not cause harmful interference, and (2) This device must accept any interference received, including interference that may cause undesired operation.

Wireless Approval For UAE In Middle East

TRA REGISTERED No: ER36153/14 or ER36153/15
DEALER No.: HONEYWELL INTERNATIONAL MIDDLE EAST – LTD – DUBAI BR

Wireless Approval For QATAR In Middle East

ictQATAR
Type Approval Reg. No.: R-4466 or R-4635

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Read Before Operating

This manual must be carefully read by all individuals who have or will have the responsibility of using, maintaining, or servicing this product. The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions. The user should understand how to set the correct parameters and interpret the obtained results.

CAUTION!

To reduce the risk of electric shock, turn the power off before removing the instrument cover. Disconnect the battery before removing sensor module for service. Never operate the instrument when the cover is removed. Remove instrument cover and sensor module only in an area known to be non-hazardous.

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Special Notes

When the instrument is taken out of the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the instrument. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.

The battery of the instrument discharges slowly even if it is turned off. If the instrument has not been charged for 5 to 7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the instrument before using it. It is also recommended to fully charge the instrument for *at least 10 hours* before first use. Refer to this User Guide's section on battery charging for more information on battery charging and replacement.

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WARNINGS

STATIC HAZARD: Clean only with damp cloth.

For safety reasons, this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

Use only RAE Systems battery packs, part numbers 059-3051-000, 059-3052-000, and 059-3054-000. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.

Do not mix old and new batteries or batteries from different manufacturers.

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.

For maximum safety, the accuracy of the instrument should be checked by exposing it to a known concentration calibration gas before each day's use.

Do not use USB/PC communication in hazardous locations.

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AVERTISSEMENT

DANGER RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipment doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

Utiliser seulement l'ensemble de batterie RAE Systems, la reference 059-3051-000 au 059-3052-000 au 059-3054-000. Cet instrument n'a pas été essayé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsique. Ne charger les batteries que dans emplacements désignés non-dangereuse.

Ne pas melanger les anciennes et les nouvelles batteries, ou bien encore les batteries de differents fabriquants.

La calibration de toute instruments de RAE Systems doivent être testé en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant de mettre en service l'instrument pour la première fois.

Pour une securite maximale, la sensibilité du l'instrument doit être verifier en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant chaque utilisation journalière.

Ne pas utiliser de connection USB/PC en zone dangereuse.

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Standard Contents

Instrument
Calibration Kit
Charging Cradle
AC/DC Adapter
Alkaline Battery Adapter
Data Cable
CD-ROM With User's Guide, Quick Start Guide, and related materials

General Information

The compact instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It monitors Volatile Organic Compounds (VOC) using a photoionization detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas-discharge lamp. Features are:

Lightweight and Compact

- Compact, lightweight, rugged design
- Built-in sample draw pump

Dependable and Accurate

- Up to 16 hours of continuous monitoring with rechargeable battery pack
- Designed to continuously monitor VOC vapor at parts-per-million (ppm) levels

User-friendly

- Preset alarm thresholds for STEL, TWA, low- and high-level peak values.
- Audio buzzer and flashing LED display are activated when the limits are exceeded.

Datalogging Capabilities

- 260,000-point datalogging storage capacity for data download to PC

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The instrument consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged case with a backlit LCD and 3 keys to provide easy user interface. It also has a built-in flashlight for operational ease in dark locations.

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Physical Description

The main components of the portable VOC monitoring instrument include:

- Three keys for user to interact with the instrument: 3 operation/programming keys for normal operation or programming
- LCD display with back light for direct readout and calculated measurements
- Built-in flashlight for illuminating testing points in dark environments
- Buzzer and red LEDs for alarm signaling whenever exposures exceed preset limits
- Charge contacts for plugging directly to its charging station
- Gas entry and exit ports
- USB communication port for PC interface
- Protective rubber cover

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Specifications

Size:	9.25" L x 3.6" W x 2.9" H
Weight:	28 oz with battery pack
Detector:	Photoionization sensor with 9.8, 10.6, or 11.7 eV UV lamp
Battery:	A 3.7V rechargeable Lithium-Ion battery pack (snap in, field replaceable, at non-hazardous location only)
	Alkaline battery holder (for 4 AA batteries)
Battery Charging:	Less than 8 hours to full charge
Operating Hours:	Up to16 hours continuous operation
Display:	Large dot matrix screen with backlight

Measurement range & resolution

Lamp	Range	Resolution
10.6 eV	0.1 ppm to 15,000 ppm	0.1 ppm
9.8 eV	0.1 ppm to 5,000 ppm	0.1 ppm
11.7 eV	0.1 ppm to 2,000 ppm	0.1 ppm

Response time (T_{90}): 2 seconds

Accuracy (Isobutylene): 10 to 2000 ppm: ±3% at calibration point.

PID Detector: Easy access to lamp and sensor for cleaning and replacement

Correction Factors: Over 200 VOC gases built in (based on RAE Systems Technical Note TN-106)

Calibration: Two-point field calibration of zero and standard reference gases

Calibration Reference: Store up to 8 sets of calibration data, alarm limits and span values

Inlet Probe: Flexible 5" tubing

Radio module: Bluetooth (2.4GHz) or RF module (433MHz, 868MHz , 915MHz, or 2.4GHz)

Keypad: 1 operation key and 2 programming keys; 1 flashlight switch

Direct Readout: Instantaneous, average, STEL, TWA and peak value, and battery voltage

Intrinsic Safety: US and Canada: Class I, Division 1, Groups A, B, C, D

Europe: ATEX (0575 Ex II 2G Ex ia IIC/IIB T4 Gb)
KEMA 07 ATEX 0127
Complies with EN60079-0:2009, EN60079-11:2007

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	IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb Complies with IEC 60079-0:2007, IEC 60079-11:2006 (IIC: 059-3051-000 Li-ion bat pack or 059-3054-000 NiMH bat pack; IIB: 059-3052-000 alkaline bat pack)
EM Interference:	Highly resistant to EMI/RFI. Compliant with EMC R&TTE (RF Modules)
Alarm Setting:	Separate alarm limit settings for Low, High, STEL and TWA alarm
Operating Mode:	Hygiene or Search mode
Alarm:	Buzzer 95dB at 30cm and flashing red LEDs to indicate exceeded preset limits, low battery voltage, or sensor failure
Alarm Type:	Latching or automatic reset
Real-time Clock:	Automatic date and time stamps on datalogged information
Datalogging:	260,000 points with time stamp, serial number, user ID, site ID, etc.
Communication:	Upload data to PC and download instrument setup from PC via USB on charging station.
Sampling Pump:	Internally integrated. Flow rate: 450 to 550 cc/min.
Wireless Network:	Mesh RAE Systems Dedicated Wireless Network (or WiFi network for WiFi-equipped instruments)
Wireless Frequency:	ISM license-free band, 902 to 907.5 MHz and 915 to 928 MHz, FCC Part 15, CE R&TTE, IEEE 802.11 b/g bands (2.4 GHz)
Modulation:	802.15.4 DSSS BPSK
RF Power (Tx):	10dBm
Temperature:	-20º C to 50º C (-4º to 122º F)

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Humidity:	0% to 95% relative humidity (non-condensing)
Housing (including rubber boot):	Polycarbonate, splashproof and dustproof Battery can be changed without removing rubber boot.

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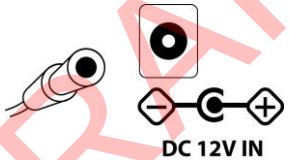
Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion battery is charged by placing the instrument in its cradle. (The battery can also be charged by placing the instrument in an AutoRAE 2 Cradle.) Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



2. Plug the AC/DC adapter into the wall outlet.
3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glow

The instrument begins charging automatically. The "Primary" LED in the cradle blinks green to indicate charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown. The cradle's LED glows continuously green.

Note: If you see the "Battery Charging Error" icon (a battery outline with an exclamation mark inside), check that the

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instrument or rechargeable battery has been set into the cradle properly. If you still receive the message, check the Troubleshooting section of this guide.

Note: If the instrument or battery has been in the cradle for more than 10 hours and you see the "Battery Charging Error" icon and a message that says, "Charging Too Long," this indicates that the battery is not reaching a full charge. Try changing the battery and make sure the contacts between the instrument (or battery) are meeting the cradle. If the message is still shown, consult your distributor or RAE Systems Technical Services.

Charging A Spare Rechargeable Battery

A rechargeable Li-ion battery can be charged when it is not inside the monitor. The charging cradle is designed to accommodate both types of charging. Contacts on the bottom of the battery meet the contacts on the cradle, transferring power without other connections, and a spring-loaded capture holds the battery in place during charging.

1. Plug the AC/DC adapter into the monitor's cradle.
2. Place the battery into the cradle, with the gold-plated contacts on top of the six matching charging pins.
3. Plug the AC/DC adapter into the wall outlet.

The battery begins charging automatically. During charging, the Secondary LED in the cradle blinks green. When charging is complete, it glows steady green.

Release the battery from the cradle by pulling it back toward the rear of the cradle and tilting it out of its slot.

Note: If you need to replace the Li-ion battery pack, replacements are available from RAE Systems. The part number is 059-3051-000.

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Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. **Remove and replace batteries only in areas known to be non-hazardous.**

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the "empty battery" icon blinks on and off once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.

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Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion battery or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

Data Protection While Power Is Off

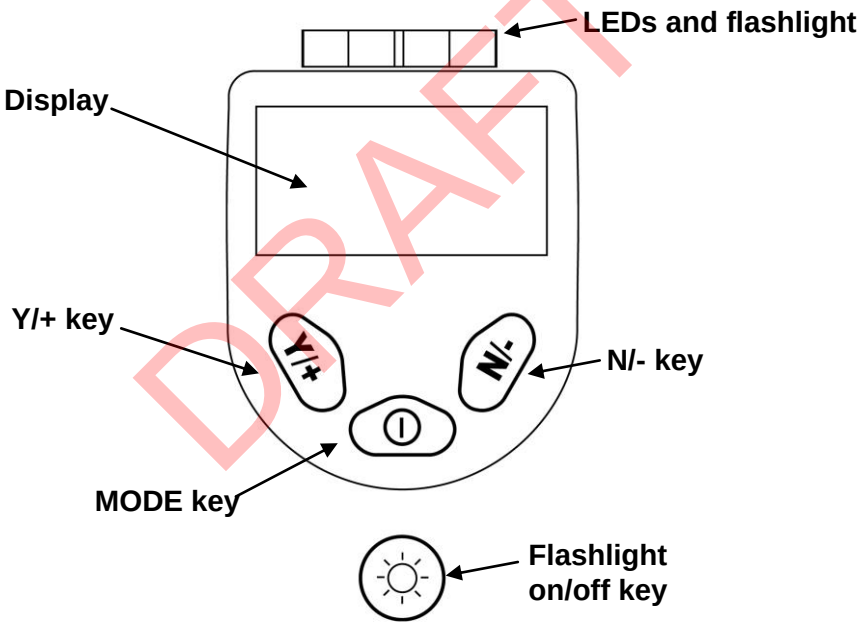
When the instrument is turned off, all the current real-time data including last measured values are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost.

User Interface

The instrument's user interface consists of the display, LEDs, an alarm transducer, and four keys. The keys are:

Y/+
MODE
N/-
Flashlight on/off

The LCD display provides visual feedback that includes the reading, time, battery condition, and other functions.



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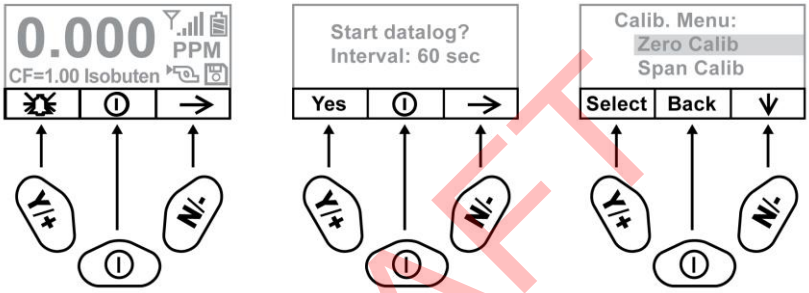
In addition to their labeled functions, the keys labeled Y/+, MODE, and N/- act as "soft keys" that control different parameters and make different selections within the instrument's menus. From menu to menu, each key controls a different parameter or makes a different selection.

Three panes along the bottom of the display are "mapped" to the keys. These change as menus change, but at all times the left pane corresponds to the [Y/+] key, the center pane corresponds to the [MODE] key, and the right pane corresponds to the [N/-] key. Here are three examples of different menus with the relationships of the keys clearly shown:

RELATIONSHIP OF BUTTONS TO CONTROL FUNCTIONS



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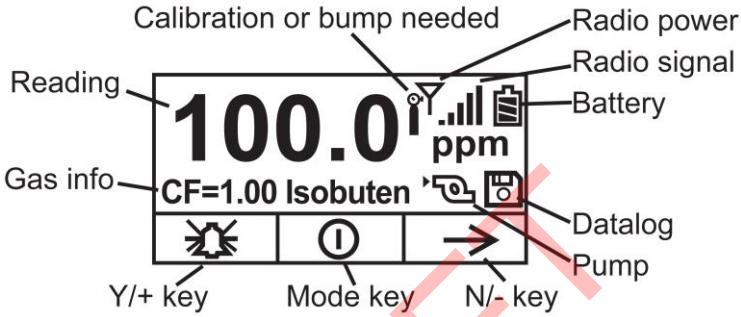
Display

The display shows the following information:



Gas info	Tells the Correction Factor and type of calibration gas
Reading	Concentration of gas as measured by the instrument
Calibration or bump needed	Dark icon indicates that calibration should be performed; light icon indicates bump should be performed
Radio power	Indicates whether radio (Mesh wireless or Bluetooth) connection is on or off
Radio signal	Indicates signal strength in 5-bar bargraph
Battery	Indicates battery level in 3 bars
Pump	Indicates that pump is working
Datalog	Indicates whether datalog is on or off
Y/+	Y/+ key's function for this screen
MODE	MODE key's function for this screen
N/-	N/- key's function for this screen

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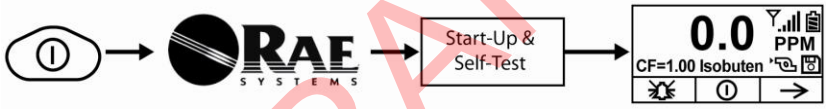
Operating The Instrument

The instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It gives real-time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the instrument is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, you should test the instrument and verify the calibration before the first use. After the instrument is fully charged and calibrated, it is ready for immediate operation.

Turning The Instrument On

1. With the instrument turned off, press and hold [MODE].
2. When the display turns on, release the [MODE] key.



The RAE Systems logo should appear first. (If the logo does not appear, there is likely a problem and you should contact your distributor or RAE Systems Technical Support.) The instrument is now operating and performs self tests. If any tests (including sensor and memory tests fail), refer to the Troubleshooting section of this guide.

Once the startup procedure is complete, the instrument shows a numerical reading screen with icons. This indicates that the instrument is fully functional and ready to use.

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Turning The Instrument Off

1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
2. Once the countdown stops, the instrument is off. Release the Mode key.
3. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Operating The Built-In Flashlight

The instrument has a built-in flashlight that helps you point the probe in dark places. Press the flashlight key to turn it on. Press it again to turn it off.

Note: Using the flashlight for extended periods shortens the battery's operating time before it needs recharging.

Pump Status

IMPORTANT!

During operation, make sure the probe inlet and the gas outlet are free of obstructions. Obstructions can cause premature wear on the pump, false readings, or pump stalling. During normal operation, the pump icon alternately shows inflow and outflow as shown here:

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During duty cycling (PID lamp cleaning), the display shows these icons in alternation:

If there is a pump failure or obstruction that disrupts the pump, you will see this icon blinking on and off:

If you see this blinking icon, consult the Troubleshooting section of this guide.

Calibration Status

The instrument displays this icon if it requires calibration:

Calibration is required (and indicated by this icon) if:

- The lamp type has been changed (for example, from 10.6 eV to 9.8 eV).
- The sensor has been replaced.
- It has been 30 days or more since the instrument was last calibrated.
- If you have changed the calibration gas type without recalibrating the instrument.

Bump Status

The instrument displays this icon if it requires a bump test:

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A bump test is required (and indicated by this icon) if:

- The defined period of time between bump tests has been exceeded (bump test overdue).
- The sensor has failed a previous bump test.
- The sensor(s) should be challenged on a periodic basis.

Policy Enforcement

The MiniRAE 3000 can be configured to enforce a facility/company's requirements that calibration and/or bump testing be performed at specified intervals, and to explicitly prompt the user that calibration/bump testing is required. Depending on how Policy Enforcement features are configured, the user may be required to perform a bump test or calibration prior to being able to use the instrument. That is, it can be set to not allow normal operation of the instrument unless calibration or bump testing is performed.

If the instrument has been bump tested and calibrated in compliance with the policy settings, a check-mark icon is included along the top of the MiniRAE 3000 screen:

If Policy Enforcement is enabled, then after startup the MiniRAE 3000 displays a screen that informs the user that the instrument requires either a bump test or a calibration. If both are required, then they are shown in sequence.

Note: Policy enforcement features are disabled by default.

Setting Policy Enforcement

You must use ProRAE Studio II to make changes to Policy Enforcement settings. You must use an AutoRAE 2 Cradle, a MiniRAE 3000 Travel Charger, or a MiniRAE 3000 Desktop Cradle. Policy violations are captured in the datalog.

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Using The Travel Charger, Desktop Charger, or AutoRAE 2 Automatic Test And Calibration System

To program a MiniRAE 3000 via an AutoRAE 2, you need ProRAE Studio II Instrument Configuration and Data Management Software, the AutoRAE 2 connected to a power source, and a USB PC communications cable.

1. Connect a USB cable between a PC with ProRAE Studio II and the AutoRAE 2 Cradle, Travel Charger, or Desktop Cradle.
2. Apply power to the AutoRAE 2 Cradle, Travel Charger, or Desktop Cradle.
3. Turn off the MiniRAE 3000 (or put the MiniRAE 3000 into AutoRAE 2 Mode or Communication Mode) and set it in the cradle.
4. Start ProRAE Studio II software on the PC.
5. Select "Administrator" and input the password (the default is "rae").
6. Click "Detect the instruments automatically" (the magnifying glass icon with the letter "A" in it). After a few seconds, the AutoRAE 2 Cradle is found and it is shown, along with its serial number.
7. Click on the icon to highlight it, and then click "Select."
8. In ProRAE Studio II, the instrument or AutoRAE 2 Cradle is shown, including its Serial Number, under "Online."
9. Expand the view to show the instrument or to show the instrument in the AutoRAE 2 Cradle by clicking the "+" to the left of the image of the AutoRAE 2 Cradle.
10. Double-click on the icon representing the MiniRAE 3000.
11. Click "Setup."
12. In the menu that now appears on the left side, click "Policy Enforcement." It is highlighted, and the Policy Enforcement pane is shown. For "Must Calibrate" and "Must Bump," you have the options of no enforcement or enforcement (including "Can't Bypass," and "Can Bypass").

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Must Calibrate. The user is prompted to calibrate the instrument when calibration is due (as set by the calibration interval). There are two programmable options:

- **Can't Bypass.** Unless calibration is performed, the instrument cannot be used, and the only option is to turn off the instrument.
- **Can Bypass.** If calibration is due but the user does not want to perform a calibration, the instrument can still be used. In this case, the instrument records that the user has bypassed the calibration requirement in a Policy Violation report.

Must Bump. The user is prompted to bump test the instrument when a bump test is due (as set by the bump test interval). There are two programmable options:

- **Can't Bypass.** Unless a bump test is performed, the instrument cannot be used, and the only option is to turn off the instrument.
- **Can Bypass.** If a bump test is due but the user does not want to perform one, the instrument can still be used. In this case, the instrument records that the user has bypassed the bump testing requirement in a Policy Violation report.

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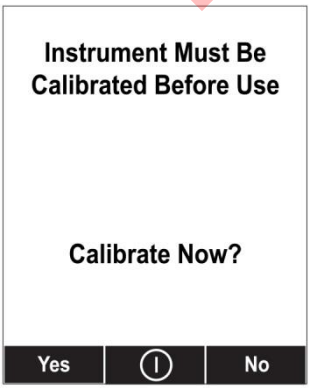
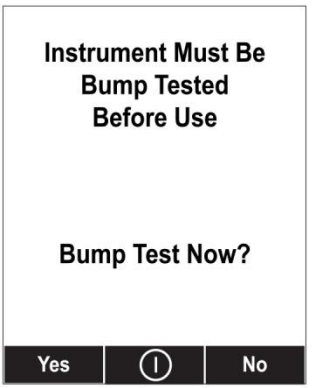
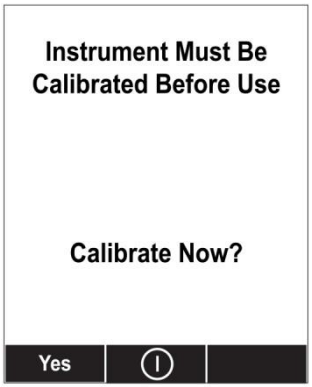
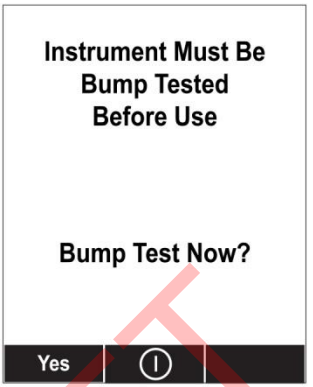
These are the screens that are shown on a MiniRAE 3000 after startup if "Can Bypass" is selected:





If "Can't Bypass" is selected, the display looks like this, and only allows the options of performing the test or shutting down:





16. Once you have made your selections in ProRAE Studio II, you must upload the changes to the instrument. Click the icon labeled "Upload all settings to the instrument."

17. A confirmation screen is shown. Click "Yes" to perform the upload, or "No" to abort.
    Uploading takes a few seconds, and a progress bar is shown. You can abort the upload by clicking "Cancel."

18. Exit ProRAE Studio II.

19. Press [Y/+] on the MiniRAE 3000 to exit Communication Mode.

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Operating Modes

Your instrument operates in different modes, depending on the model and its factory default settings. In some cases, you can change modes using a password and using the instrument's navigation. In other cases, you must use ProRAE Studio software.

The default setting for your instrument is:

User Mode: Basic
Operation Mode: Hygiene

This is outlined in detail on page 83.

The other options, covered later in this guide, are:

User Mode: Advanced (page 86)
Operation Mode: Hygiene

User Mode: Advanced (page 90)
Operation Mode: Search

Using ProRAE Studio allows access to other options. In addition, Diagnostic Mode (page 91) is available for service technicians.

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Basic User Level/Hygiene Mode (Default Settings)

The instrument is programmed to operate in Basic User Level/Hygiene Mode as its default. This gives you the most commonly needed features while requiring the fewest parameter adjustments.

Pressing [N/-] steps you from one screen to the next, and eventually return to the main display. If you do not press a key within 60 seconds after entering a display, the instrument reverts to its main display.

Note: While viewing any of these screens, you can shut off your instrument by pressing [MODE].

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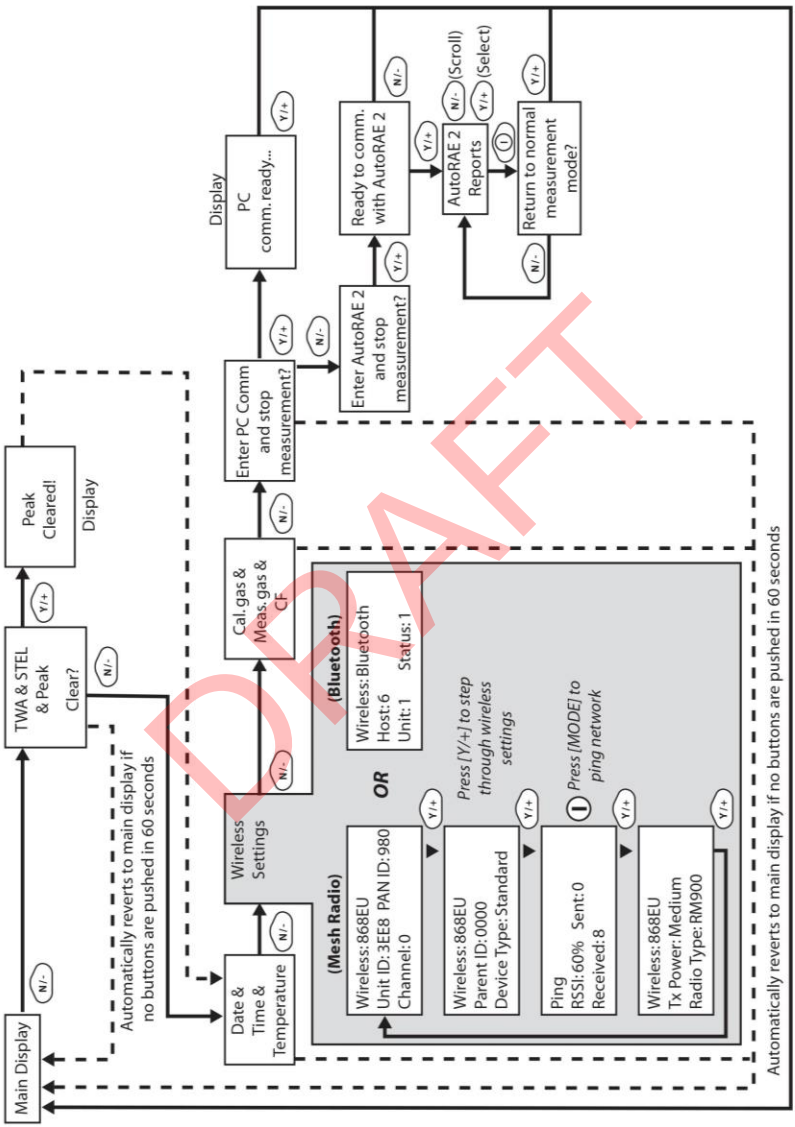

Note: Dashed line indicates automatic progression.

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After the instrument is turned on, it runs through the start-up menu. Then the message "**Please apply zero gas…**" is displayed.

At this point, you can perform a zero air (fresh air) calibration. If the ambient air is clean, you can use that. Otherwise, use a cylinder of zero air. Refer to Zero Calibration on page 44 for a more detailed description of zero calibration.

Start zero calibration by pressing Start. You see the message "Zeroing…" followed by a 30-second countdown.

Note: You can press [MODE] to quit, bypassing the zero air calibration.

When zero calibration is complete, you see the message:

Zeroing is done!

Reading = 0.0 ppm

The instrument is now sampling and collecting data.

Note: At the Average & Peak, Date & Time & Temperature, Calibration Gas & Measurement Gas & Correction Factor, and PC Communications screens, the instrument automatically goes to the main display after 60 seconds if you do not push a key to make a selection.

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Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, or pump stall.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display
Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

* Hygiene mode only. In Search mode, the number of beeps per second (1 to 7) depends upon the concentration of the sampled gas. Faster rates indicate higher concentrations.

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Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
MiniRAE 3000	100	ppm	50	100	10	25

Testing The Alarm

You can test the alarm whenever the main (Reading) display is shown. Press [Y/+], and the audible and visible alarms are tested.

Integrated Sampling Pump

The instrument includes an integrated sampling pump. This diaphragm-type pump that provides a 450 to 550 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8" inside diameter to the gas inlet port of the instrument, this pump can pull in air samples from 100' (30 m) away horizontally or vertically.

Note: In Search Mode, the pump turns on when a sample measurement is started, and turns off when the sample is manually stopped.

If liquid or other objects are pulled into the inlet port filter, the instrument detects the obstruction and immediately shuts down the pump. The alarm is activated and a flashing pump icon is displayed.

You should acknowledge the pump shutoff condition by clearing the obstruction and pressing the [Y/+] key while in the main reading display to restart the pump.

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Backlight

The LCD display is equipped with an LED backlight to assist in reading the display under poor lighting conditions.

Datalogging

During datalogging, the instrument displays a disk icon to indicate that datalogging is enabled. The instrument stores the measured gas concentration at the end of every sample period (when data logging is enabled). In addition, the following information is stored: user ID, site ID, serial number, last calibration date, and alarm limits. All data are retained (even after the unit is turned off) in non-volatile memory so that it can be down- loaded at a later time to a PC.

Datalogging event

When Datalogging is enabled, measurement readings are being saved. These data are stored in "groups" or "events." A new event is created and stored each time the instrument is turned on and is set to automatic datalogging, or a configuration parameter is changed, or datalogging is interrupted. The maximum time for one event is 24 hours or 28,800 points. If an event exceeds 24 hours, a new event is automatically created. Information, such as start time, user ID, site ID, gas name, serial number, last calibration date, and alarm limits are recorded.

Datalogging sample

After an event is recorded, the unit records a shorter form of the data. When transferred to a PC running ProRAE Studio, this data is arranged with a sample number, time, date, gas concentration, and other related information.

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Auto/Manual/Snapshot Datalogging

The instrument has three datalog types:

Auto	Default mode. Collects datalog information when the instrument is sampling.
Manual	Datalogging occurs only when the instrument's datalogging is manually started (see page 63 for details).
Snapshot	Datalogs only during snapshot (single-event capture, initiated by pressing [MODE]) sampling. See page 65 for details.

Note: You can only choose one datalog type to be active at a time.

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Accessories

The following accessories are included with the instrument:

- An AC Adapter (Battery Charger)
- Alkaline battery adapter
- External Filter
- Organic Vapor Zeroing kit

Hard-case kits also include these accessories:

- Calibration adapter
- Calibration regulator and Flow controller

Standard Kit & Accessories

AC Adapter (Battery Charger)

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WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in area known to be non-hazardous.

Ne charger les batteries que dans emplacements designés non-dangereuses.

A battery charging circuit is built into the instrument cradle. It only needs a regular AC to 12 VDC adapter (wall-mount transformer, part number 500-0114-000) to charge the instrument.

To charge the battery inside the instrument:

1. Power off the instrument.
2. Connect the AC adapter to the DC jack on the instrument's cradle. If the instrument is off, it automatically turns on.
3. While charging, the display message shows "Charging." The Primary LED on the cradle flashes green when charging.
4. When the battery is fully charged, the LED changes to glowing green continuously, and the message "Fully charged" appears on the

display. If there is a charging error, the LED glows red continuously.

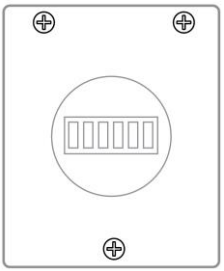
A completely discharged instrument can be charged to full capacity within 8 hours. Batteries drain slowly even if an instrument is off. Therefore, if the instrument has been in storage or has not been charged for several days or longer, check the charge before using it.

The factory-supplied battery is designed to last for 16 hours of normal operation (no alarm), for a new battery under the optimum circumstances. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), its capacity will be significantly reduced.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:



1. Remove the three Philips-head screws to open the compartment in the adapter.
2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
3. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

1. Remove the Li-ion battery pack from the instrument by sliding the tab and tilting out the battery.
2. Replace it with the alkaline battery adapter
3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The

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internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument. If you try to charge an alkaline batteries installed in the instrument, the instrument's display will say, "Alkaline Battery," indicating that it will not charge the alkaline batteries.

Note: When replacing alkaline batteries, dispose of old ones properly.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

External Filter

The external filter is made of PTFE (Teflon®) membrane with a 0.45 micron pore size to prevent dust or other particles from being sucked into the sensor manifold, which would cause extensive damage to the instrument. It prolongs the operating life of the sensor. To install the external filter, simply connect it to the instrument's inlet tube.

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Optional Accessories

Calibration Adapter

The calibration adapter for the instrument is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the instrument and the tubing to the gas regulator on the gas bottle.

Calibration Regulator

The Calibration Regulator is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the instrument during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per min.). Alternatively, a demand-flow regulator or a Tedlar gas bag may be used to match the pump flow precisely.

Organic Vapor Zeroing Kit

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the instrument.

AutoRAE 2 Automatic Test & Calibration System

The AutoRAE 2 Automatic Test and Calibration System for RAE Systems portable gas monitors makes compliance with monitor test and calibration requirements as easy as pressing a button. Simply cradle the monitor and the system will take care of all calibration, testing, and recharging.

The AutoRAE 2 is a flexible, modular system that can be configured to meet your calibration requirements effectively and efficiently. An AutoRAE 2 system can be as simple as a single cradle deployed in standalone mode to calibrate one instrument at a time, or as powerful as a networked, controller-based system supporting ten monitors and five distinct calibration gas cylinders.

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Standard Two-Point Calibration (Zero & Span, Optional Bump)

The following diagram shows the instrument's calibrations in Basic/Hygiene mode.

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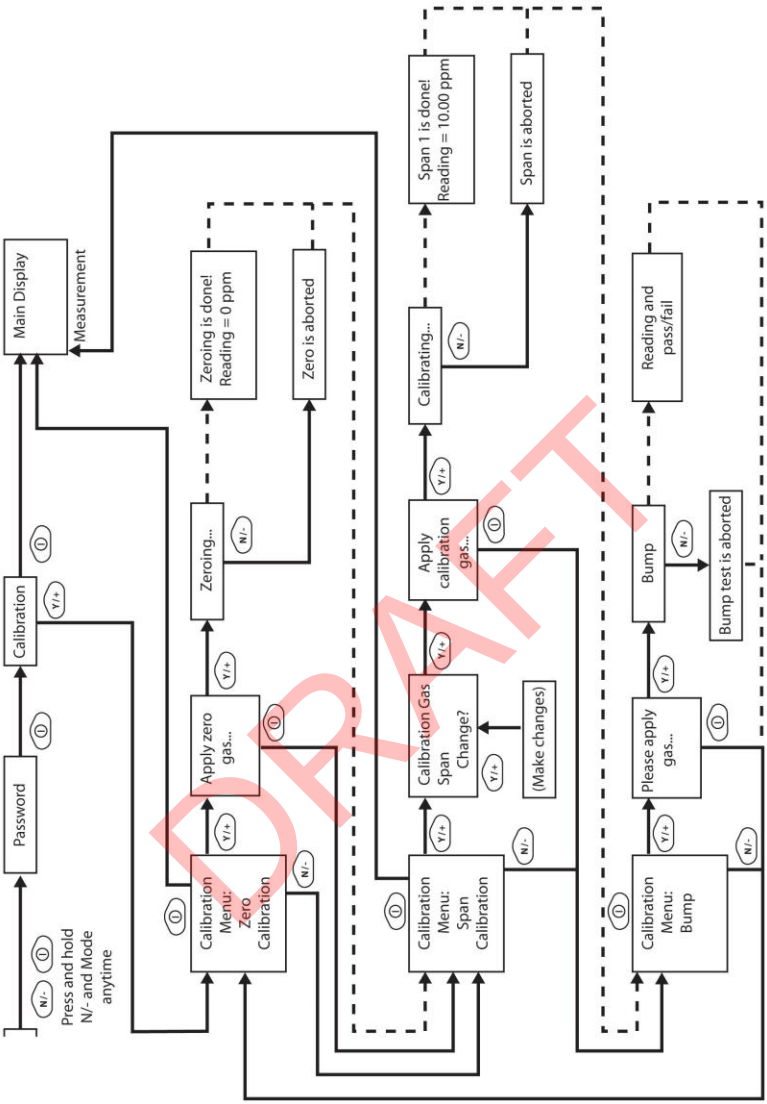
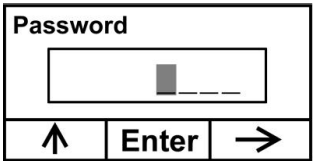


Note: Dashed line indicates automatic progression.

Entering Calibration

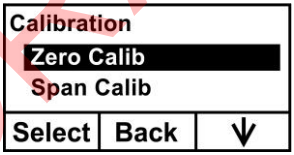
1. Press and hold [MODE] and [N/-] until you see the Password screen.



2. In Basic User Level, you do not need a password to perform calibrations. Instead of inputting a password, enter calibration by pressing [MODE].

   Note: If you inadvertently press [Y/+] and change any of the numbers, simply press [MODE] and you will be directed to the calibration menu.

The Calibration screen is now visible with Zero Calibration highlighted.



These are your options:

- Press [Y/+] to select the highlighted calibration (Zero Calib or Span Calib).
- Press [MODE] to exit calibration and return to the main display and resume measurement.
- Press [N/-] to toggle the highlighted calibration type.

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Zero (Fresh Air) Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the instrument to a "fresh" air source such as from a cylinder or Tedlar bag (optional accessory). The "fresh" air is clean, dry air without organic impurities and an oxygen value of 20.9%. If such an air cylinder is not available, any clean ambient air without detectable contaminants or a charcoal filter can be used.

At the Zero Calibration menu, you can proceed to perform a Zero calibration or bypass Zero calibration and perform a Span calibration. You may also go back to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to start calibration.
- Press [MODE] to quit and return to the main calibration display.

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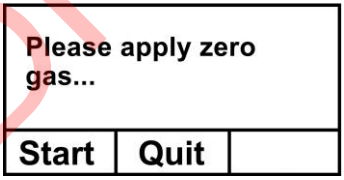
If you have pressed [Y/+] to enter Zero calibration, then you will see this message:



1. Turn on your Zero calibration gas.
2. Press [Y/+] to start calibration.

   Note: At this point, you may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu, highlighted for Span calibration.

3. Zero calibration starts a 30-second countdown and displays this message:

   Zeroing...

During the zeroing process, the instrument performs the Zero calibration automatically and does not require any action on your part.

Note: To abort the zeroing process at any time and proceed to Span calibration, press [N/-] at any time while zeroing is being performed. You will see a confirmation message that says "Zero aborted!" and then the Span calibration menu appears.

When Zero calibration is complete, you see this message:

Zeroing is done!
Reading = 0.0 ppm

The instrument will then show the Calibration menu on its display, with Span Calib highlighted.

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Span Calibration

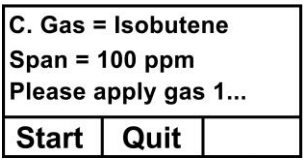
This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:



1. Turn on your span calibration gas.
2. Press [Y/+] to initiate calibration.

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Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will abort the span calibration and take you directly to the Calibration menu for Zero calibration.

3. Span calibration starts and displays this message:

   Calibrating...

During the Span calibration process, there is a 30-second countdown and the instrument performs the Span calibration automatically. It requires no actions on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu appears. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you see a message similar to this (the value is an example only):

Span 1 is done!
Reading = 100.0 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

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Exiting Two-Point Calibration In Basic User Level

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings…

The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

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Three-Point Calibration

For enhanced accuracy, it is possible to perform a second Span calibration in addition to the Zero and Span calibrations outlined in the previous section. Your instrument first must be set to allow this third calibration. This requires using ProRAE Studio software and a PC, as well as a higher concentration of calibration gas.

Note: Once the third calibration is set, you do not need to use ProRAE Studio to allow future 3-point calibrations. Also, you can only disable 3-point calibration capability by using ProRAE Studio again.

Perform the Zero and Span calibrations. After the first Span calibration (Span 1) is completed, the display a second Span calibration (Span 2) can be performed. The process is identical to the first calibration. As in the Span 1 calibration, you may exit and return to the Zero calibration screen if you choose not to perform this calibration or to abort it.

Note: If a bump test is available, it appears after the last calibration in the menu. See "Two-Point Calibration," page 38, for details. Also, refer to page 53 for details on how to perform a bump test.

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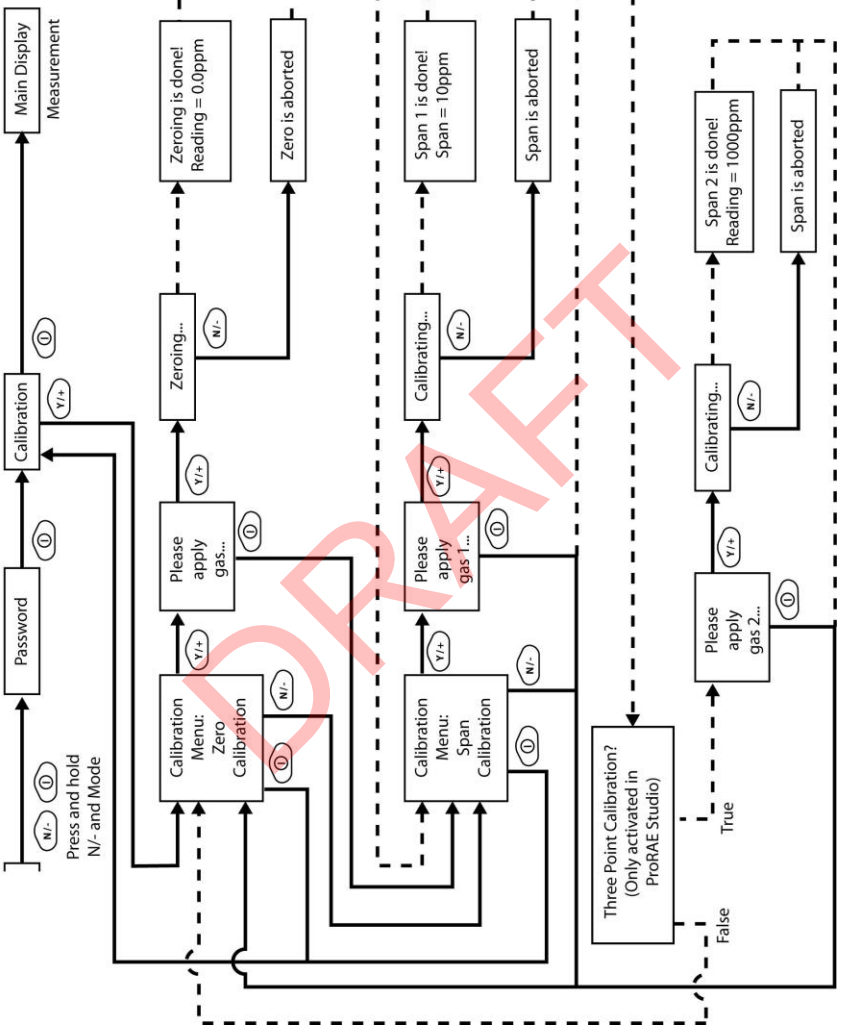

Note: Dashed line indicates automatic progression.

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Span 2 Calibration

A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure.

Note: This gas should be of a higher concentration than the gas used for Span 1 calibration.

Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span 2 calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:

Please apply gas...

4. Turn on your span calibration gas.
5. Press [Y/+] to initiate calibration.

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Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu for Zero calibration.

6. Span calibration starts a 30-second countdown and displays this message:

   Calibrating...

During the Span calibration process, the instrument performs the Span calibration automatically and does not require any action on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu will appear. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you will see a message similar to this (the value shown here is for example only):

Span 2 is done!
Reading = 1000 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

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Exiting Three-Point Calibration

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings…

The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Bump Test

RAE Systems recommends that a bump test be conducted prior to each day's use. The purpose of a bump test is to ensure that the instrument's sensors respond to gas and all the alarms are enabled and functional.

- The MiniRAE 3000 must be calibrated if it does not pass a bump test when a new sensor is installed, after sensor maintenance has been performed, or at least once every 180 days, depending on use and sensor exposure to poisons and contaminants.
- Calibration and bump test intervals and procedures may vary due to national legislation and company policy.

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To perform a bump test (functional challenge), follow these steps:

1. Select "Bump."
2. Install the calibration adapter and connect it to a source of calibration gas.
3. Verify that the displayed calibration value meets the concentration specified on the gas cylinder.
4. Start the flow of calibration gas.
5. Press [Y/+] to start the bump test.
6. You can abort the calibration at any time during the countdown by pressing [N/-].

7. If the calibration is not aborted, the display shows reading and then tells you whether the bump test passed or failed. If the bump test failed, then it automatically advances to the Calibration screen.

Important!

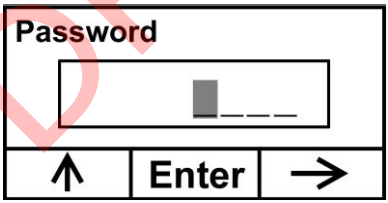
Anytime a bump test fails, you should perform a full calibration of the instrument.

Programming Mode

Programming Mode can be entered from either Hygiene Mode or Search Mode. If the current user mode is Basic, you must provide a 4-digit password to enter.

Entering Programming Mode

1. Press and hold [MODE] and [N/-] until you see the Password screen.



2. Input the 4-digit password:

- Increase the number from 0 through 9 by pressing [Y/+].
- Step from digit to digit using [N/-].
- Press [MODE] when you are done.

If you make a mistake, you can cycle through the digits by pressing [N/-] and then using [Y/+] to change the number in each position.

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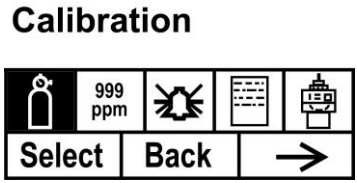
Note: The default password is 0000.

When you have successfully entered Programming Mode, you see this screen:



Note: The password can only be changed by connecting the instrument to a PC running ProRAE Studio software. Follow the instructions in ProRAE Studio to change it.

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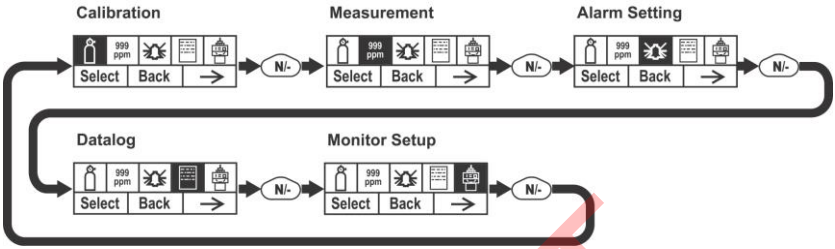
The Calibration label is shown and its icon is highlighted, but you can press [N/-] to step from one programming menu to the next, with the name of the menu shown at the top of the display and the corresponding icon highlighted. As you repeatedly press [N/-], the selection moves from left to right, and you see these screens:



Note: When you reach Monitor Setup and press [N/-], the menu cycles back to Calibration.

Programming Mode Menus

The Programming Mode allows anyone with the password to change the instrument's settings, calibrate the instrument, modify the sensor configuration, enter user information, etc. Programming Mode has five menus. Each menu includes several sub-menus to perform additional programming functions.

This table shows the menus and sub-menus:

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Calibration	**Measurement**	**Alarm Setting**	**Datalog**	**Monitor Setup**
Zero Calibration	Meas. Gas	High Alarm	Clear Datalog	Radio Power
Span Calibration	Meas. Unit	Low Alarm	Interval	Op Mode
Bump		STEL Alarm	Data Selection	Site ID
		TWA Alarm	Datalog Type	User ID
		Alarm Mode		User Mode
		Buzzer & Light		Date
				Time
				Pump Duty Cycle
				Pump Speed
				Temperature Unit
				Language
				Real Time Protocol
				Power On Zero
				Unit ID
				LCD Contrast
				Lamp ID
				PAN ID
				Mesh Channel
				Mesh Interval

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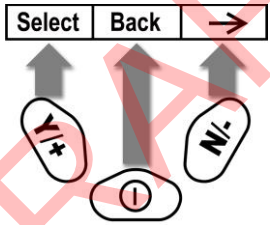
Once you enter Programming Mode, the LCD displays the first menu, Calibration. Each subsequent menu is accessed by pressing [N/-] repeatedly until the desired menu is displayed. To enter a sub-menu of a menu, press [Y/+].

Exiting Programming Mode

To exit Programming Mode and return to normal operation, press [MODE] once at any of the programming menu displays. You will see "Updating Settings…" as changes are registered and the mode changes.

Navigating Programming Mode Menus

Navigating through the Programming Mode menus is easy and consistent, using a single interface format of "Select," "Back" and "Next" at the top level. The three control buttons correspond to these choices as shown:



Note: Pressing [MODE] in the Programming Mode's top level causes the instrument to exit Programming Mode and return to monitoring.

The three keys perform the following functions in Programming Mode:

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Provides a "no" response to a question

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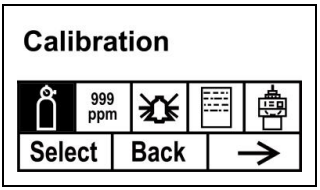
Calibration

Two types of calibration are available: Zero (fresh air) and Span.



Select Zero or Span Calibration by pressing [N/+]. Once your choice is highlighted, press [Y/+].

Zero Calibration

The procedure for performing a zero calibration is covered on page 41.

Span Calibration

The procedure for performing a basic span calibration is covered on page 41.

Bump

The procedure for performing a bump calibration is covered on page 53.

A bump test can be performed either manually or using the AutoRAE 2 Automatic Test and Calibration System. When a bump test is done manually, the instrument makes a pass/fail decision based on sensor performance, but the user still has the responsibility to make sure all the alarms are enabled and functional.

Note: Bump testing and calibration can be performed using an AutoRAE 2 Automatic Test & Calibration System. An AutoRAE 2 bump test takes care of both the sensor and alarm tests. Consult the AutoRAE 2 User's guide for details.

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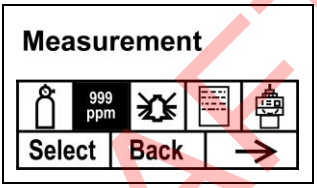
IMPORTANT!

If the instrument does not pass a bump test, perform a full calibration. If calibration also fails, the PID sensor or lamp may require cleaning or replacement. If the instrument repeatedly fails to calibrate, turn it off and refer it for servicing.

Measurement

The sub-menus for Measurement are Measurement Gas and Measurement Unit.



Meas. Gas

Measurement gases are organized in four lists:

- My List is a customized list of gases that you create. It contains a maximum of 10 gases and can only be built in ProRAE Studio on a PC and transferred to the instrument. **Note:** The first gas in the list is always isobutylene (it cannot be removed from the list).
- Last Ten is a list of the last ten gases used by your instrument. The list is built automatically and is only updated if the gas selected from Custom Gases or Library is not already in the Last Ten. This ensures that there is no repetition.
- Gas Library is a library that consists of all the gases found in RAE Systems' Technical Note TN-106 (available online at www.raesystems.com).
- Custom Gases are gases with user-modified parameters. Using ProRAE Studio, all parameters defining a gas can be modified,

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including the name, span value(s), correction factor, and default alarm limits.

1. Scroll through each list by pressing [N/-].
2. Press [Y/+] to select one (My List, Last Ten, Gas Library, or Custom Gases).
3. Once you are in one of the categories, press [N/-] to scroll through its list of options and [Y/+] to select one. (If you press [MODE], you exit to the next submenu.)
4. Press [Y/+] to save your choice or [N/-] to undo your selection.

Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

Meas. Unit

Standard available measurement units include:

Abbreviation	Unit	MiniRAE 3000
ppm	parts per million	Yes
ppb	parts per billion	
mg/m3	milligrams per cubic meter	Yes
ug/m3	micrograms per cubic meter	

- Scroll through the list by pressing [N/-].
- Select by pressing [Y/+].
- Save your selection by pressing [Y/+] or undo your selection by pressing [N/-].

Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

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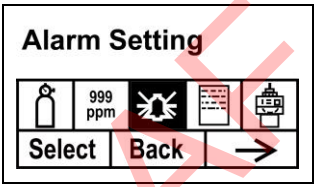
Alarm Setting

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn of the alarm condition.

An alarm signal summary is shown on page 33.

In this menu, you can change the High and Low alarm limits, the STEL limit, and the TWA. Press [Y/+] to to enter the Alarm Setting menu.
Note: All settings are shown in ppb (parts per billion), or µg/m^3 (micrograms per cubic meter), depending on your setting.



1. Scroll through the Alarm Limit sub-menu using the [N/-] key until the display shows the desired limit to be changed (High Alarm, Low Alarm, STEL Alarm, and TWA Alarm)
2. Press [Y/+] to select one of the alarm types. The display shows a flashing cursor on the left-most digit of the previously stored alarm limit.
3. Press [Y/+] to increase each digit's value.
4. Press [N/-] to advance to the next digit.
5. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

Press [MODE] when you are done.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

When all alarm types have been changed or bypassed, press [MODE] to exit to the Programming Menu.

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High Alarm

You can change the High Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the High Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.
When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

Press [Y/+] to save the changes.
Press [N/-] to undo the changes and revert to the previous settings.

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Low Alarm

You can change the Low Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the Low Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

STEL Alarm

You can change the STEL Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the STEL Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

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TWA Alarm

You can change the TWA (time-weighted average) Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the TWA Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices:

- Save
- Undo

You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

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Alarm Mode

There are two selectable alarm modes:

Auto Reset	When the alarm condition is no longer present, the alarm stops and automatically resets itself.
Latch	When the alarm is triggered, you can manually stop the alarm. The latched setting only controls alarms for High Alarm, Low Alarm, STEL Alarm, and TWA alarm. **Note:** To clear an alarm when the instrument is set to "Latched," press [Y/+] when the main (Reading) display is shown.

1. Press **[N/-]** to step from one alarm type to the other.
2. Press **[Y/+]** to select an alarm type.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

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Buzzer & Light

The buzzer and light alarms can be programmed to be on or off individually or in combination. Your choices are:

- Both on
- Light only
- Buzzer only
- Both off

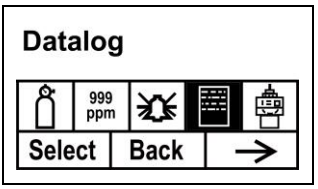
1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selections, press [MODE].

   You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

   - Press [Y/+] to save the changes.
   - Press [N/-] to undo the changes and revert to the previous settings.

Datalog

The instrument calculates and stores the concentration and ID of each sample taken. In the datalog sub-menu, a user can perform the tasks and functions shown below.



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1. Scroll through the Datalog sub-menu using the [N/-] key until the display shows the desired parameter to be changed:

   Clear Datalog
   Interval
   Data Selection
   Datalog Type

2. Press [Y/+] to make your selection. Exit by pressing [MODE] for Back.

Clear Datalog

This erases all the data stored in the datalog.

Note: Once the datalog is cleared, the data cannot be recovered.

Press [Y/+] to clear the datalog. The display asks, "Are you sure?"

- Press [Y/+] if you want to clear the datalog. When it has been cleared, the display shows "Datalog Cleared!"
- Press [N/-] if you do not want to clear the datalog.

The display changes, and you are taken to the next sub-menu, Interval.

Interval

Intervals are shown in seconds. The default value is 60 seconds. The maximum interval is 3600 seconds.

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

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Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Data Selection

Data Selection allows you to select which types of data are stored and made available when you offload your datalog to a computer via ProRAE Studio software.

You can choose any or all of three types of data (you must choose at least one):

- Average
- Maximum
- Minimum

1. Press [N/-] to step from one option to the next. The highlighter indicates your choice.
2. Press [Y/+] to toggle your selection on or off (the check box indicates "on" with an "X").
3. When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

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Datalog Type

The instrument has three datalog types:

Auto	Default mode. Collects datalog information when the instrument is sampling.
Manual	Datalogging occurs only when the instrument's datalogging is manually started (see below for details).
Snapshot	Datalogs only during single-event capture sampling.

Note: You can only choose one datalog type to be active at a time.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.

Press [N/-] to undo the changes and revert to the previous settings.

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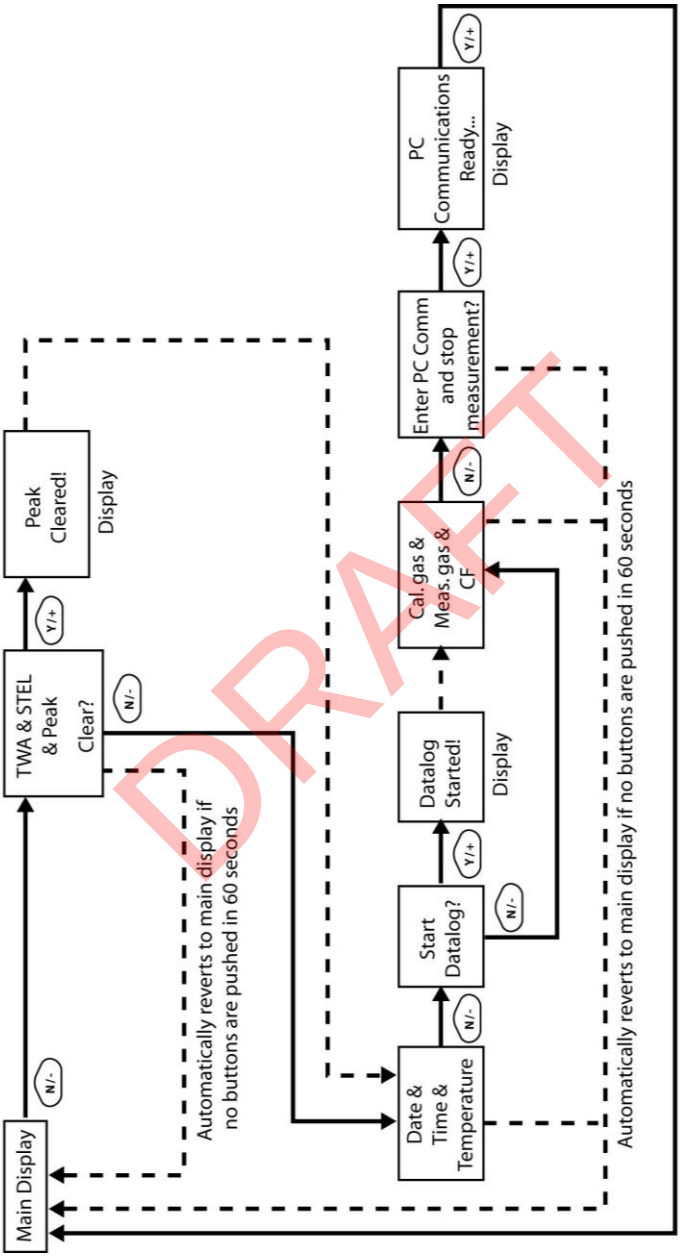
Manual Datalog

When the instrument is set to Manual Datalog, you turn datalogging on and off by stepping through the displays from the Main Display, and then pressing the keys to select datalog on/off functions.

- When you reach the screen that says "Start Datalog?" press [Y/+] to start it. You see "Datalog Started," confirming that datalogging is now on.

When you reach the screen that says "Stop Datalog?" press [Y/+] to stop it. You see "Datalog Stopped," confirming that datalogging is now off.



Note: Dashed line indicates automatic progression.

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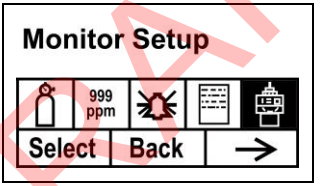
Snapshot Datalog

When the instrument is in Snapshot datalogging mode, it captures a single "snapshot" of the data at the moment of your choosing. Whenever the instrument is on and it is set to Snapshot, all you have to do is press [MODE] each time you want to capture a snapshot of the data at that instant.

When you send the data to a computer using ProRAE Studio, the data snapshots are uniquely identified by time and other parameters.

Monitor Setup

Many settings can be accessed in this menu, including setting the date and time and adjusting the pump's on/off duty cycle.



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Radio Power

The radio connection can be turned on or off.

1. Press [N/-] to step from one option to the next (on or off).
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates that the option is selected).
3. When you have completed your selection, press [MODE].

- Press [Y/+] to accept the new radio setting (on or off).
- Press [N/-] to discard the change and move to the next sub-menu.

Op Mode

Under Monitor Setup is "Op Mode."

Press [Y/+] to select.

You see two options (one is highlighted):

Hygiene
Search

The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

1. Select Hygiene or Search by pressing [N/-]. The highlighting changes from one to the other each time you press [N/-].
2. Press [Y/+] to select that mode for the instrument.
3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

Site ID

Enter an 8-digit alphanumeric/character Site ID in the programming mode. This Site ID is included in the datalog report.

1. Press [Y/+] and the display shows the current site ID. Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).
   Note: The last four digits must be numerals.
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

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Repeat this process until all eight digits of the new site ID are entered.

Press [MODE] to exit.

If there is any change to the existing site ID, the display shows "Save?" Press [Y/+] to accept the new site ID. Press [N/-] to discard the change and move to the next sub-menu.

User ID

Enter an 8-digit alphanumeric User ID in the programming mode. This User ID is included in the datalog report.

1. Press [Y/+] and the display shows the current User ID. Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all eight digits of the new User ID are entered.

Press [MODE] to exit.

If there is any change to the existing User ID, the display shows "Save" Press [Y/+] to accept the new site ID. Press [N/-] to discard (undo) the change and move to the next sub-menu.

User Mode

The instrument has two user modes:

Basic Basic users can only see and use a basic set of functions.
Advanced Advanced users can see all screens and perform all available functions.

Note: The default value for User Mode is Basic.

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To change the User Mode:

1. Press [N/-] to step from one option to the next. The highlighting changes each time you press [N/-].
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
4. Press [Y/+] to accept the new User Mode. Press [N/-] to discard the change and move to the next sub-menu.

Date

The Date is expressed as Month/Day/Year, with two digits for each.

1. Press [Y/+] and the display shows the current date. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all six digits of the new date are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

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Time

The Time is expressed as Hours/Minutes/Seconds, with two digits for each. The time is in 24-hour (military) format.

1. Press [Y/+] and the display shows the current time. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all six digits of the new time are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Pump Duty Cycle

The pump's duty cycle is the ratio of its on time to off time. The duty cycle ranges from 50% to 100% (always on), and the period is 10 seconds. Therefore, a duty cycle of 60% means that the pump is on for 6 seconds and off for four seconds. Duty cycling is employed by the instrument to clean the PID. A lower duty cycle has a greater effect on keeping the PID clean than a higher duty cycle.

Important! Pump duty cycling is interrupted when the instrument senses a gas. The pump's duty cycle is disabled when the measurement is greater than the 2ppm threshold and is re-enabled when the reading falls below 90% of the threshold (1.8 ppm).

1. Press [Y/+] to increase the value.
2. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new duty cycle value.
   - Press [N/-] to undo the change and move to the next sub-menu.

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Pump Speed

The pump can operate at two speeds, high and low. Running at low speed is quieter and conserves a small amount of power. There is almost no difference in sampling accuracy.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new temperature unit.
   - Press [N/-] to undo the change and move to the next sub-menu.

Temperature Unit

The temperature display can be switched between Fahrenheit and Celsius units.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new temperature unit.
   - Press [N/-] to undo the change and move to the next sub-menu.

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Language

English is the default language, but other languages can be selected for the instrument.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save your new language choice.
   - Press [N/-] to undo it and return to the previous language selection.

Real Time Protocol

Real Time Protocol is the setting for data transmission.

The choices are:

P2M (cable) Point to multipoint. Data is transferred from the instrument to multiple locations using a wired connection. Default data rate: 19200 bps.

P2P (cable) Point to point. Data is transferred only between the instrument and one other location, such as a computer. Default data rate: 9600 bps.

P2M (wireless) Point to multipoint, wireless. Data is transferred wirelessly and can be received by multiple receivers.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new real-time communications protocol.
   - Press [N/-] to undo the change and move to the next sub-menu.

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Power On Zero

When Power On Zero is on, the instrument performs a zero calibration when it is turned on.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the change.
   - Press [N/-] to discard the change and move to the next sub-menu.

Unit ID

This three-digit number keeps data separated by instrument when more than one instrument is used in a network. If multiple sensing units are attempting to communicate with the same Host, then the units must all have a different Unit ID.

1. Press [Y/+] to step through all 10 numerals (0 to 9). If you pass the numeral you want, keep pressing [Y/+]. After it counts up to 9, it starts counting up from 0 again.
2. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all three digits of the Unit ID are entered.

3. Press [MODE] when you are done.
   - Press [Y/+] to save the change.
   - Press [N/-] to discard the change and move to the next sub-menu.

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LCD Contrast

The display's contrast can be increased or decreased from its default setting. You may not need to ever change the default setting, but sometimes you can optimize the display to suit extreme temperature and ambient brightness/darkness conditions.

- The minimum value is 20.
- The maximum value is 60.

1. Press [Y/+] to increase the value or [N/-] to decrease the value.
2. Press [MODE] to save your selection.
   - Press [Y/+] to save your new contrast value.
   - Press [N/-] to undo it and return to the previous value.

Lamp ID

The instrument must be set to the correct lamp value in order to function correctly. Always match the value that was installed in your instrument from the factory or the value of the PID lamp you are replacing.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].

PAN ID

The MiniRAE 3000 and any other devices that you want to interconnect wirelessly must have the same PAN ID. You can set the PAN ID in the instrument or through ProRAE Studio II.

1. Press [N/-] to advance through the digits from left to right.
2. Press [Y/+] to] to advance through the numbers (1, 2, 3, etc.).
3. Press [MODE] to register your choice when you are done.

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Mesh Channel

Note: For mesh radio modems operating at 868MHz, only channel 0 is available. For other frequencies, channels 1 through 10 are allowed.

1. Press [Y/+] to increase the number and [N/-] to advance to the next digit.
2. After moving to the last digit and making changes, press [MODE].
    - Press [Y/+] to save the change.
    - Press [N/-] to undo the change.

Mesh Interval

Set the time interval at which the instrument's mesh radio sends out a signal. This can range from once every 10 seconds to once every four minutes (240 seconds). The transmission frequency is user-adjustable, but a rate of at least once every 30 seconds is recommended. **Note:** Shorter intervals reduce battery life.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make a selection.
3. When you are done, press [MODE].

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Hygiene Mode

The instrument usually operates in Hygiene Mode, which provides basic functionality. However, it is possible to operate it in a second mode called Search Mode. Here are the primary differences:

Hygiene Mode: Automatic measurements, continuously running and datalogging, and calculates additional exposure values.

Search Mode: Manual start/stop of measurements and display of certain exposure values.

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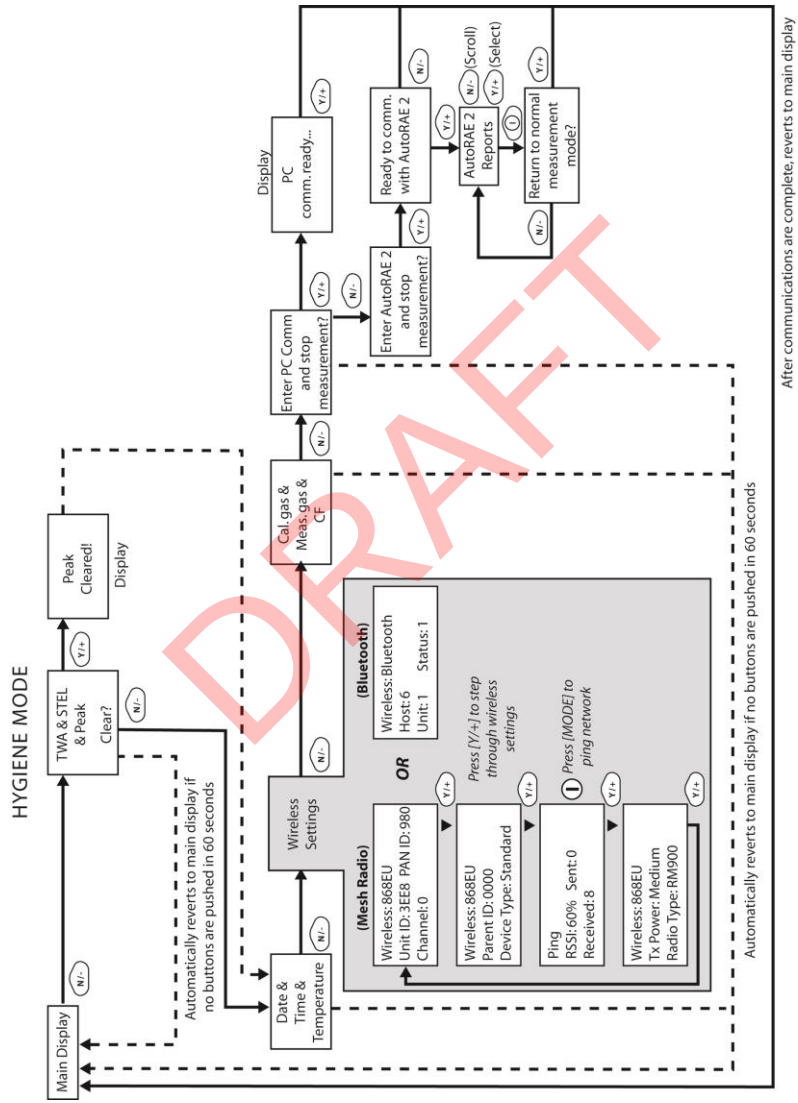
Basic User Level & Hygiene Mode

The default setting is navigated in the following way:



Note: Dashed line indicates automatic progression.

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Pressing [N/-] steps you from screen to screen. Options include clearing the Peak value and turning on the instrument's PC Communications for data transfer to a PC.

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Entering Search Mode From Hygiene Mode

In order to change the instrument's operational mode from Hygiene Mode to Search Mode, you must enter the password-protected Programming Mode:

1. Hold [MODE] and [N/-] until you see the password screen.
2. Use [Y/+] to increment to the number you want for the first digit. (If you pass by the desired number, press [Y/+] until it cycles through to 0 again. Then press [Y/+] until you reach the desired number.)
3. Press [N/-] to advance to the next digit.
4. Again press [Y/+] to increment the number.
5. Press [N/-] to advance to the next digit.

Continue the process until all four numbers of the password have been input. Then press [MODE] to proceed.

The screen changes to icons with the label "Calibration."

1. Press [N/-] to advance to "Monitor Setup."
2. Press [Y/+] to select Monitor Setup.

Under Monitor Setup, you will see "Op Mode."

Press [Y/+] to select.

You will see:

Hygiene
Search

The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

1. Select Hygiene or Search by pressing [N/-].
2. Press [Y/+] to place the instrument into the selected mode.

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3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

Advanced User Level (Hygiene Mode Or Search Mode)

The User Mode called Advanced User Level allows a greater number of parameters to be changed than Basic User Level. It can be used with either of the Operation Modes, Hygiene Mode or Search Mode.

Advanced User Level & Hygiene Mode

With the instrument in Operation Mode: Hygiene Mode, enter User Mode: Advanced User Level (refer to the section called Monitor Mode for instructions).

Once you are in Advanced User Level and Hygiene Mode together, you can change the calibration reference and measurement gas, in addition to performing normal monitoring functions.

Pressing [N/-] progresses through the screens, while pressing [Y/+] selects options. Pressing [MODE] makes menu choices when it is shown for "Done" or "Back." Pressing and holding [Mode] whenever the circle with a vertical line in the middle is shown activates the countdown to shutoff.

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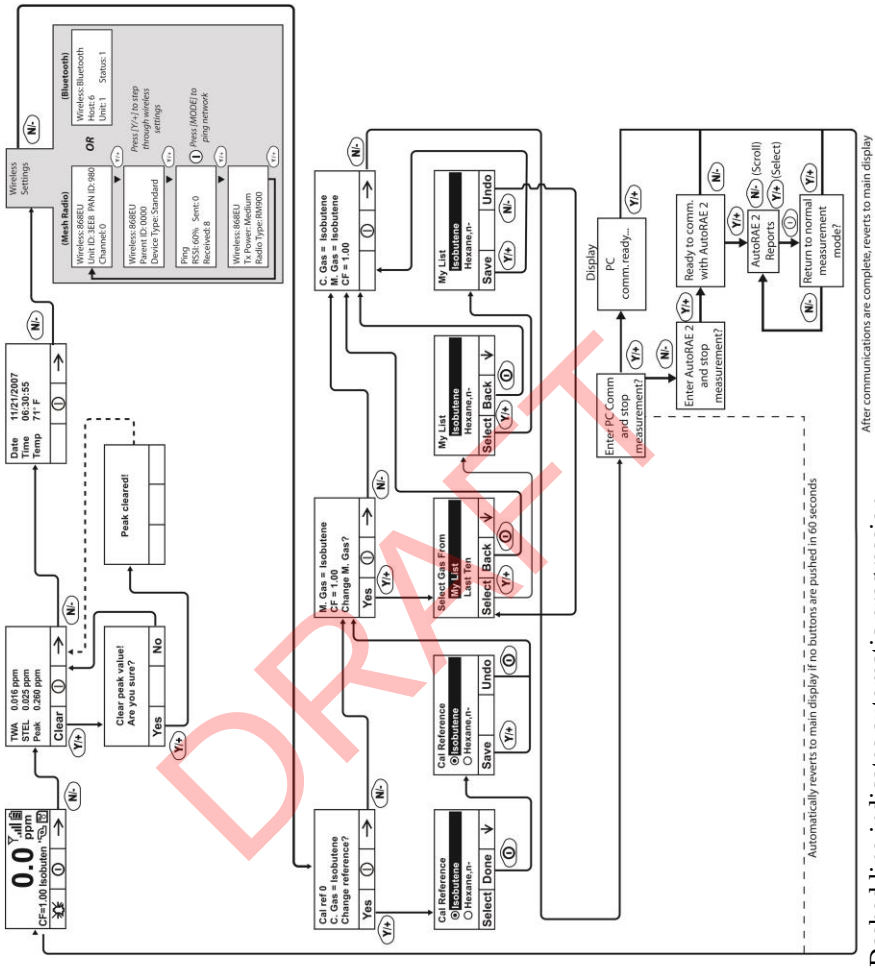

Note: Dashed line indicates automatic progression.

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Basic User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Basic User Level (refer to the section called User Mode for instructions).

When the instrument is in Search Mode, it only samples when you activate sampling. When you see the display that says, "Ready…Start sampling?" press [Y/+] to start. The pump turns on and the instrument begins collecting data. To stop sampling, press [N/-] while the main display is showing. You will see a new screen that says, "Stop sampling?" Press [Y/+] to stop sampling. Press [N/-] if you want sampling to continue.

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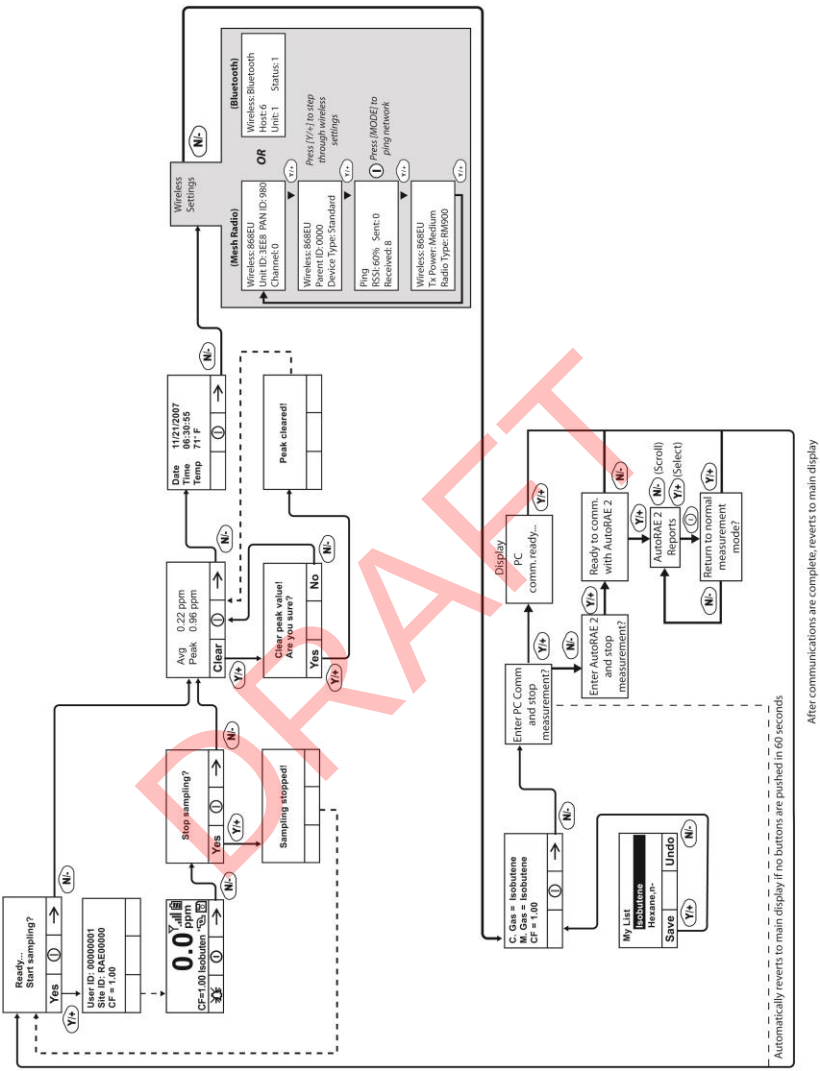

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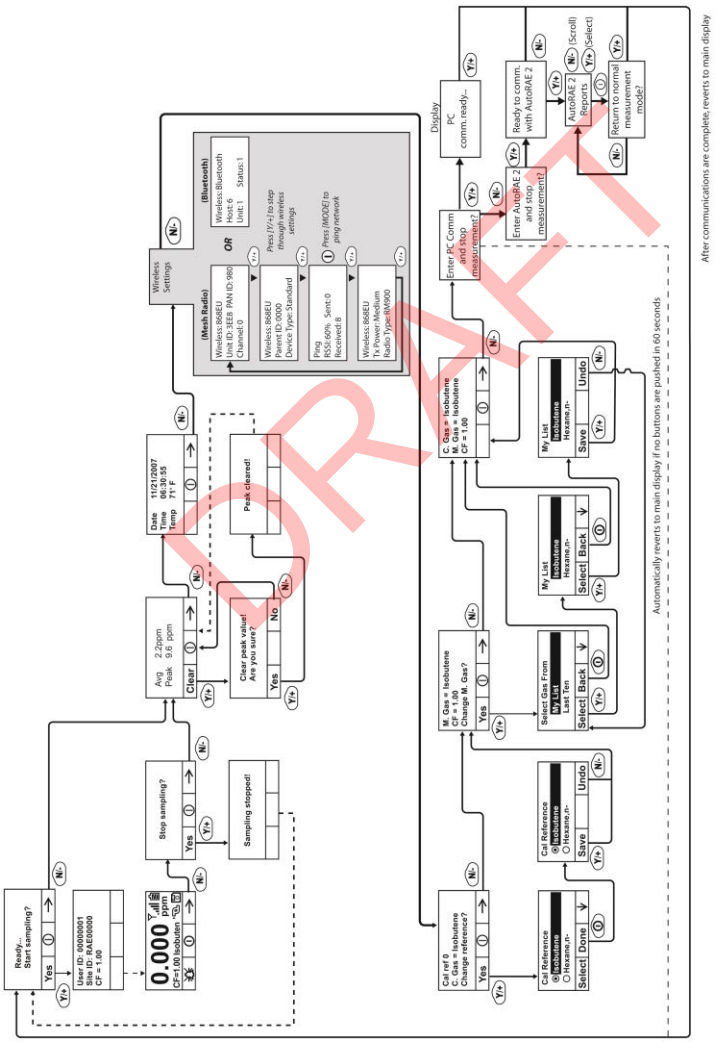
Note: Dashed line indicates automatic progression.

Advanced User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Advanced User Level (refer to the section called Monitor Mode for instructions). Operation is similar to Basic User Level & Sampling Mode, but now allows you to change calibration and measurement reference gases. Refer to the section on measurement gases on page 60 for more details.



Note: Dashed line indicates automatic progression.

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Diagnostic Mode

IMPORTANT! Diagnostic Mode is designed for servicing and manufacturing, and therefore is not intended for everyday use, even by advanced users. It provides raw data from sensors and about settings, but only allows adjustment of pump stall parameters, which should only be changed by qualified personnel.

Note: If the instrument is turned on in Diagnostic Mode and you switch to User Mode, datalog data remains in raw count form. To change to standard readings, you must restart the instrument.

Entering Diagnostic Mode

Note: To enter Diagnostic Mode, you must begin with the instrument turned off.

Press and hold [Y/+] and [MODE] until the instrument starts.

The instrument goes through a brief startup, and then displays raw data for the PID sensor. These numbers are raw sensor readings without calibration. The instrument is now in Diagnostic Mode.

Note: In Diagnostic Mode, the pump and lamp are normally on.

You can enter Programming Mode and calibrate the instrument as usual by pressing both [MODE] and [N/-] for three seconds.

You can enter Monitoring Mode by pressing [MODE] and [Y/+] together for three seconds.

Once the instrument is started up in Diagnostic Mode, you can switch between Diagnostic Mode and Monitoring Mode by pressing and holding [MODE] and [Y/+] simultaneously for two seconds.

In Diagnostic mode, you can step through parameter screens by pressing [MODE].

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Adjusting The Pump Stall Threshold

If the gas inlet is blocked but the pump does not shut down, or the pump shuts down too easily with a slight blockage, the pump stall threshold value may be set too high or too low.

Use the following steps to adjust the pump stall threshold:

Pump High

In Diagnostic Mode, press the [MODE] key until "Pump High" is displayed. The display shows the maximum, minimum, and stall values for the pump at its high speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

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Pump Low

In Diagnostic Mode, press the [MODE] key until "Pump Low" is displayed. The display shows the maximum, minimum, and stall values for the pump at its low speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that

number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

Exiting Diagnostic Mode

You can exit Diagnostic Mode and go directly to Programming Mode or Monitor Mode as outlined above, or you can exit Diagnostic Mode completely.

To exit Diagnostic Mode so that it cannot be re-entered without a restart:

Shut down the instrument. When it is off, restart it by holding the [MODE] key. Diagnostic Mode cannot be entered until the instrument is restarted as outlined in "Entering Diagnostic Mode."

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Transferring Data To & From A Computer

Once you have connected your instrument cradle to the PC, you can can transfer data, including a download of the datalog to the computer and updates of firmware to the instrument (should this ever be necessary).

Downloading The Datalog To A PC

1. Connect the data cable to the PC and the cradle.
2. Place the instrument into its cradle. The charging LED should be illuminated.
3. Start ProRAE Studio on your PC.
4. From ProRAE Studio, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the instrument.
6. To receive the datalog in the PC, select "Downlog Datalog."
7. When you see "Unit Information," click OK.

During the data transfer, the display shows a progress bar.

When the transfer is done, you will see a screen with the datalog information. You can now export this datalog for other use or printing.

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Uploading Firmware To The instrument From A PC

Uploading new firmware to your instrument requires connecting the instrument and PC. Follow these steps to make the connection:

1. Connect the data cable to the PC and the cradle.
2. Place the instrument into its cradle. The charging LED should be illuminated.
3. Start RAEProgrammer 7000 on your PC.
4. From RAEProgrammer 7000, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the instrument.
6. Select Operation → Download Firmware.

Once communication is established, follow the instructions that accompany RAEProgrammer 7000 and the firmware to upload the new firmware to your instrument.

Note: Check for the latest updates to ProRAEProgrammer 7000 at www.raesystems.com.

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Maintenance

The major maintenance items of the instrument are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

Note: Maintenance should be performed by qualified personnel only.

NOTE: The printed circuit board of the instrument is connected to the battery pack even if the power is turned off. **Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the instrument**. **Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.**

Battery Charging & Replacement

When the display shows a flashing empty battery icon, the battery requires recharging. It is recommended to recharge the instrument upon returning from fieldwork. A fully charged battery runs a instrument for 16 hours continuously. The charging time is less than 8 hours for a fully discharged battery. The battery may be replaced in the field (in areas known to be non-hazardous), if required.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. **Remove and replace battery only in areas known to be non-hazardous.**

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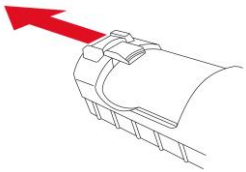
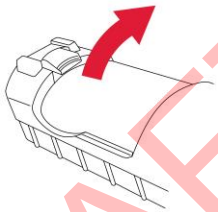
Replacing The Li-ion Battery

1. Turn off the instrument.
2. Located on the rear of the instrument is a battery tab. Slide it down to unlock the battery.



3. Remove the battery pack from the battery compartment by tilting it out.



4. Replace a fully charged spare battery pack inside the battery compartment. Make sure the battery pack is oriented properly inside the compartment.
5. Slide the capture tab back up to its locked position.

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Replacing The Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:

1. Remove the three Philips-head screws to open the compartment.
2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
3. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

1. Remove the Li-ion battery pack from the battery compartment by sliding the tab and tilting out the battery.
2. Replace it with the alkaline battery adapter
3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument.

Note: When replacing alkaline batteries, dispose of old ones properly.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

Note: The internal charging circuit is designed to prevent charging to alkaline batteries.

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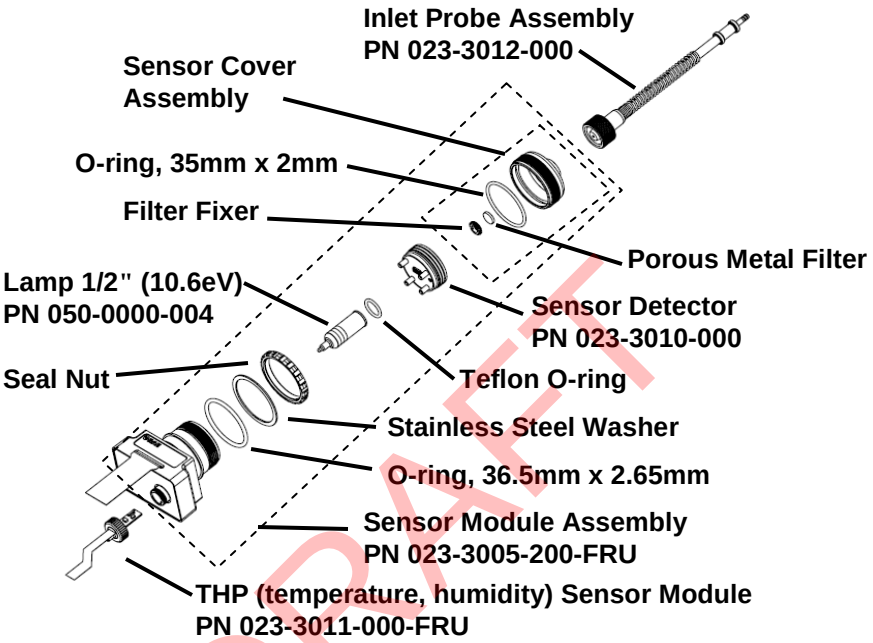
PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown below.



Sensor Components

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Note: The cleaning procedure is not normally needed. Clean the PID sensor module, the lamp and the lamp housing only if:

1. The reading is inaccurate even after calibration.
2. The reading is very sensitive to air moisture.
3. A liquid has been sucked into the unit and damaged the unit.

Use of the external filter helps to prevent contamination of the sensor.

To access the sensor components and lamp, gently unscrew the lamp-housing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull it straight out. A slight, gentle rocking motion helps release the sensor.

Cleaning The PID Sensor

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.
Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode "fingers" should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

Cleaning The Lamp Housing Or Changing The Lamp

If the lamp does not turn on, the instrument will display an error message to indicate replacement of the lamp may be required.

1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

   CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.

2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
3. Reinstall the PID sensor module.
4. Tighten the Lamp Housing Cap.

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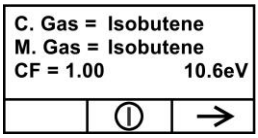
Determining The Lamp Type

The monitor can accommodate three lamp values: 10.6eV (standard), 9.8eV, and 11.7eV. Always make sure you are using the correct lamp value and that the instrument is set to use that lamp.

Also, when the monitor is running, the lamp type is shown along with the calibration and measurement gas and Correction Factor:



Note: This screen can be accessed from the reading screen by pressing [N/-] four times.

You can manually determine the lamp type, too:

1. Turn off the instrument and remove the lamp. Now look at the serial number. The following identify the lamp type:

    - 10.6eV SN: 106 2Nxxxxx
    - 9.8eV SN: 098 2Nxxxxx
    - 11.7eV SN: 117 2Nxxxxx

Programming The Lamp ID

The correct measurement gas library is used by the instrument when you ensure that the right lamp value is programmed.

To manually select the Lamp ID:

1. Enter the Programming menu.
2. Select Monitor Setup.
3. Scroll down and select the Lamp ID sub-menu.
4. Press [N/-] to scroll down to the desired Lamp ID.
5. Press [Y/+] to select.
6. Press [MODE] to select Done.
7. Select "Save."
8. Return to the main menu.

Recalibrate the instrument before returning it to service.

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Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.

If the pump is not working properly, refer the instrument to qualified service personnel for further testing and, if necessary, pump repair or replacement.

Cleaning The Instrument

Occasional cleaning with a soft cloth is recommended. Do not use detergents or chemicals.

Visually inspect the contacts at the base of the instrument, on the battery, and on the charging cradle to make sure they are clean. If they are not, wipe them with a soft, dry cloth. Never use solvents or cleaners.

Ordering Replacement Parts

If you need replacement parts, contact your local RAE Systems distributor. A list is available online:

http://www.raesystems.com

In the U.S., you can order sensors, replacement batteries, and other accessories online at:

http://istore.raesystems.com/

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Special Servicing Note

If the instrument needs to be serviced, contact either:

1. The RAE Systems distributor from whom the instrument was purchased; they will return the instrument on your behalf.

   or

2. The RAE Systems Technical Service Department. Before returning the instrument for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the instrument is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

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Troubleshooting

Problem	Possible Reasons & Solutions	
Cannot turn on power after charging the battery	**Reasons:**	Discharged battery. Defective battery.
	Solutions:	Charge or replace battery.
Lost password	**Solutions:**	Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally High	**Reasons:**	Dirty filter. Dirty sensor module. Excessive moisture and water condensation. Incorrect calibration.
	Solutions:	Replace filter. Blow-dry the sensor module. Calibrate the unit.
Reading abnormally Low	**Reasons:**	Dirty filter. Dirty sensor module. Weak or dirty lamp. Incorrect calibration.
	Solutions:	Replace filter. Remove Calibration Adapter. Calibrate the unit. Check for air leakage.
Buzzer Inoperative	**Reasons:**	Bad buzzer.
	Solutions:	Check that buzzer is not turned off. Call authorized service center.

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Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on. Replace UV lamp

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Technical Support

To contact RAE Systems Technical Support Team:

Monday through Friday, 7:00AM to 5:00PM Pacific (US) Time
Phone (toll-free): +1 888-723-4800
Phone: +1 408-952-8461
Email: tech@raesystems.com

RAE Systems Contacts

RAE Systems by Honeywell
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: 408.952.8200
Toll-Free: 888.723.4800
Fax: 408.952.8480

E-mail (technical support): RAE-tech@honeywell.com
Web Site: www.raesystems.com

WORLDWIDE SALES OFFICES

USA/Canada 1.877.723.2878
Europe +800.333.222.44/+41.44.943.4380
Middle East +971.4.450.5852
China +86.10.5885.8788-3000
Asia Pacific +852.2669.0828

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Controlled Part of Manual

Intrinsic Safety:
US and Canada: Class I, Division 1, Groups A,B,C,D T4

Europe: ATEX (0575 Ex II 2G Ex ia IIC/IIB T4 Gb)
KEMA 07 ATEX 0127
Complies with EN60079-0:2009, EN60079-11:2007
IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb
Complies with IEC 60079-0:2007, IEC 60079-11:2006

Temperature: -20º C to 50º C (-4º to 122º F)

Humidity: 0% to 95% relative humidity (non-condensing)

Basic Operation

Turning The Instrument On

1. With the instrument turned off, press and hold [MODE].
2. When the display turns on, release the [MODE] key.

The instrument is now operating and performs self tests. Once the self tests are complete, the display shows a graph or numerical gas reading. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
2. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

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Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, pump stall, or when the datalog memory is full.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display

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Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
ppbRAE 3000	10	ppm	10	25	10	25
MiniRAE 3000	100	ppm	50	100	10	25
MiniRAE Lite	100	ppm	50	100	10	25
UltraRAE 3000	100	ppm	50	100	10	25

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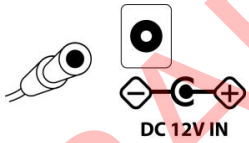
Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion/NiMH battery is charged by placing the instrument in its cradle. Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



2. Plug the AC/DC adapter into the wall outlet.
3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glows.

Note: To release the instrument, press down and tilt the top out of the cradle and lift up.

The instrument begins charging automatically. The LED on the front of the cradle marked "Primary" blinks during charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown and the Primary LED on the cradle glows continuously green.

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Note: A spare Li-ion battery (059-3051-000) or NiMH(059-3054-000) can be charged by placing it directly in the charging port on the back of the cradle. It can be charged at the same time as the instrument. Press the battery in place, sliding it slightly toward the front of the cradle. This locks it in the cradle. To release the battery, slide it forward again and tilt it up.

Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. Remove and replace batteries only in areas known to be non-hazardous.

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Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the battery icon blinks once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.

Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion, NiMH, or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in an area known to be non-hazardous.

Replacing Rechargeable Li-Ion or NiMH Battery

Caution: Turn off the instrument before removing or replacing the battery.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500).

Do not mix old and new batteries or different type batteries.

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Troubleshooting

Problem	Possible Reasons & Solutions	
Cannot turn on power after charging the battery	**Reasons:**	Discharged battery. Defective battery.
	Solutions:	Charge or replace battery.
Lost password	**Solutions:**	Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally High	**Reasons:**	Dirty filter. Dirty sensor module. Excessive moisture and water condensation. Incorrect calibration.
	Solutions:	Replace filter. Blow-dry the sensor module. Calibrate the unit.
Reading abnormally Low	**Reasons:**	Dirty filter. Dirty sensor module. Weak or dirty lamp. Incorrect calibration.
	Solutions:	Replace filter. Remove Calibration Adapter. Calibrate the unit. Check for air leakage.
Buzzer Inoperative	**Reasons:**	Bad buzzer.
	Solutions:	Check that buzzer is not turned off. Call authorized service center.

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Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on. Replace UV lamp

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RAE Systems by Honeywell
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: 408.952.8200
Toll-Free: 888.723.4800
Fax: 408.952.8480

E-mail (technical support): RAE-tech@honeywell.com
Web Site: www.raesystems.com

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WORLDWIDE SALES OFFICES

USA/Canada 1.877.723.2878
Europe +800.333.222.44/+41.44.943.4380
Middle East +971.4.450.5852
China +86.10.5885.8788-3000
Asia Pacific +852.2669.0828

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cox environmental

CES-SOP-05: Field Documentation

All field documentation will be recorded per the following Alaska Department of Environmental Conservation (ADEC) guidance documents:

- **Field Sampling Guidance (ADEC, 2017)**
- **Underground Storage Tanks Procedures Manual: Guidance for Treatment of Petroleum-Contaminated Soil and Groundwater and Standard Sampling Procedures (ADEC, 2017)**
- **Site Characterization Work Plan and Reporting Guidance for Investigation of Contaminated Sites (ADEC, 2017)**

CES personnel will document all field readings, sample locations, and field observations in a field record or log book. Logbooks or field records must be bound books that are permanently assigned to a specific project. Field forms and camera may also be used for field documentation in a variety of activities. Field forms include borehole logs, well construction, well sampling, site safety and health plan forms, etc. It is not necessary to duplicate information recorded on a field form into the logbook. All logbooks and field form entries must be printed legibly using a waterproof pen. All field forms must be completed in full on a daily basis. Entries to the field notebooks must include the following items if applicable:

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- Project name/Site ID/Client/Page Number
- Date
- Weather, site conditions, and other salient observations
- Full name of on-site personnel, affiliations and project title e.g., team leader (including all visitors)
- Daily objectives
- Time and location of activities
- Field observations and comments
- Deviations from the CSP site-specific approved work plan
- Photographic log (photographic name, roll or frame number, description of photograph, date, and time)
- Site sketches with reference to north direction, sample and field screening locations and depths, and on-site groundwater flow direction
- Survey and location (latitude and longitude coordinates when possible)
- All field measurements (e.g. leak check results, geochemical parameters, field screening results)
- Daily equipment calibrations and maintenance
- Sample record (sample identification, date, time, media, number of samples, and location)
- Cleanup or remediation activities (system performance, system calibration or maintenance record, excavation activities and volume of material removed)
- Waste tracking (when, how much, destination)
- Soil boring logs will include: blow counts, visual or olfactory observations, field screening readings, soil type, soil moisture, groundwater depth if encountered

CES personnel will correct erroneous field record or log book entries with a single line through the error. Do not erase incorrect information. Date and initial revised entries. Logbooks and field forms will be kept in the project file when complete or when not in use. Include complete copies of all field notes and field records in reports submitted to ADEC.

ATTACHMENT D

PFAS SPECIFIC SAMPLING INFORMATION

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Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

PFAS contamination poses site characterization, sampling, and analytical challenges. PFAS have unique chemical and physical properties and they often occur in complex mixtures that can change over time. At environmental investigation sites, very low concentrations of several different PFAS must be sampled and analyzed. Many materials used in the course of environmental investigation can potentially contain PFAS. There is limited published research or guidance on how certain materials used by field staff affect sample results.

ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies regarding PFAS. This fact sheet describes methods for evaluating PFAS in the environment, including:

- site characterization considerations
- sampling precautions
- laboratory analytical methods

USEPA has compiled an online resource for PFAS that includes topics such as policy and guidance, chemistry and behavior, occurrence, toxicology, site characterization, and remediation technologies (USEPA 2017h). The National Groundwater Association (NGWA) has also published a resource on PFAS that includes information about sampling and analytical methods (NGWA 2017).

2 Site Characterization Considerations

The purpose of site characterization is to understand the sources of contamination, site-specific contaminant fate and transport, and potential exposures and risks posed by a site. The site characterization techniques and study principles for PFAS-contaminated sites are generally the same as for any other site contaminated by hazardous substances. General site investigation principles and techniques will not be covered in this fact sheet, as these are well described in many existing guidance documents (for example, ASTM International 2011, 2013a, 2013b, 2014a, 2014b; Intergovernmental Data Quality Task Force (IDQTF) 2005; USEPA 1987, 1988a, 2000a, 2006c, 2013a, 2016i).

The unique chemical characteristics, uses, and transport mechanisms of PFAS should be accounted for when characterizing a contaminated site. PFAS sources (including ambient sources) pose many challenges, including their frequent occurrence as mixtures, the role of precursors, and the persistence and mobility of PFAS relative to other environmental contaminants.

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2.1 Sources and Site Identification

The *Environmental Fate and Transport* fact sheet contains conceptual site models, including descriptions and figures, for four different common source scenarios. Phase 1 site characterization investigations (ASTM 2013c) may miss the potential for PFAS contamination at a site because these chemicals historically were not considered hazardous. Comparing timelines of site history (for example, processes, layout, chemical use, and release history) with the timeline of PFAS use and with existing drinking water data (for example, the UCMR3 data [USEPA 2017f]) can be helpful in determining source identification. A solid understanding of historical uses and the past presence of PFAS is critical to identifying PFAS that may have been released at a site. See the *History and Use* fact sheet for more information.

Another challenge is that commercial products and industrial releases may consist of complex PFAS mixtures that change over time through fate and transport mechanisms and may include unidentified PFAS. Changes in manufacturing practices as well as formula modifications also complicate the source identification. When characterizing source areas, there is often a focus on only perfluoroalkyl acids (PFAAs), particularly perfluorooctane sulfonate (PFOS) and perfluorooctanoate (PFOA), which are the current chemicals of concern. These and other chemicals of concern were often released as part of original PFAS mixtures, but also may be transformation products of PFAA precursors. The focus on PFAAs means that significant portions of the total PFAS contamination might be missed, leading to underestimates of plume life expectancy for groundwater and mass flux as well as PFAS contaminant mass.

The variation in mixtures of PFAS, associated with different processes and products, may provide signatures that help identify source areas and distinguish between multiple sources. However, careful analysis is needed to distinguish between signatures associated with differing sources and those due to environmental partitioning or multiple releases over time.

Knowledge of PFAS fate, transport, and mode of release is essential to placing sampling locations. Some PFAS released at aqueous film-forming foam (AFFF) training or application sites or by industrial air emissions may result in large, diffuse areas of soil contamination (rather than point sources) that act as sources of groundwater contamination. Air emissions

from industries using PFAS may result in releases to soil and surface water, with subsequent infiltration to groundwater (Davis et al. 2007; Shin et al. 2011).

2.2 Development of Initial Conceptual Site Model (CSM)

Conceptual site models for four different common source scenarios are included in the *Environmental Fate and Transport* fact sheet. These may be useful in developing a site-specific CSM. The CSM should include sources, site history, transport and exposure pathways, and receptor identification for a specific site. Any information pertaining to potential off-site PFAS contributors, such as landfills, wastewater treatment facilities, industrial sites, fire training areas and other sources, should be considered when determining possible secondary sources of PFAS.

2.2.1 Atmospheric, Geologic, and Hydrogeologic Framework

As with all contaminated sites, characterization relies upon an adequate understanding of the geology and hydrogeology of the site. Several PFAS, including the PFAAs of current regulatory concern, are relatively mobile in groundwater. Studies have reported both biotic and abiotic transformations of some polyfluorinated substances, referred to as precursors, which may form PFAAs. However, there is no evidence that PFAAs degrade or otherwise transform under ambient environmental conditions. Thus, PFAS plumes in groundwater may travel for several miles from the original source. At sites with highly permeable, low-organic matter soils, PFAS plumes can be extensive.

Partitioning behavior of perfluorocarboxylates (PFCAs) and perfluorosulfonates (PFSAs) has been studied more than that of other PFAS. PFCAs and PFSAs are organic anions at all environmentally relevant pH values and tend to be mobile in groundwater (Xiao et al. 2015). However, these compounds, especially those with longer carbon chains, often associate with the organic carbon fraction of soil or sediment (Higgins and Luthy 2006; Guelfo and Higgins 2013) when present in the saturated zone. See the *Environmental Fate and Transport* fact sheet for more information.

At sites where PFAS are detected in surface water, the CSM should address the potential for PFAS transport by surface water and infiltration of the PFAS to groundwater in areas downstream of the site. Some PFAS are highly soluble and resistant to breakdown in the environment, which means they may be transported significant distances in surface water (Awad et al. 2011; Kwadijk, Kotterman, and Koelmans 2014). In Minnesota, PFAS-contaminated surface water moving through a natural and manmade drainage system was found to have infiltrated to groundwater in multiple locations (losing streams, lakes, ditches, and stormwater ponds) creating large, discreet areas of groundwater contamination several miles from the original source areas (ATSDR 2008; MDH 2017).

A thorough understanding of the geology and hydrogeology of a site (including groundwater-surface water interactions and air-surface water interactions) can make selection of sampling locations more efficient and reduce the number of required samples. Without careful preparation, multiple, and sometimes redundant, field efforts can make site characterization costly.

2.2.2 Investigation Strategies

Many PFAS sites consist of releases that occurred decades before PFAS were regulated. As a result, contaminant plumes have had years to develop, and in some cases, stabilize. Therefore, site characterization should not necessarily proceed the same way as for newer sites with more recent releases. At these sites, sampling begins near the source area and steps outward to determine extent. For PFAS releases, however, contamination may have occurred in areas upgradient of drinking water sources, thus drinking water supply sampling should be a top priority to ensure that human receptors are protected. Data from private drinking water supply wells may be useful in determining the extent of contaminant plumes, if the well construction and characteristics information are available.

After evaluating drinking water, soils should be characterized to determine the three-dimensional extent of soil and groundwater contamination. Soil and groundwater sampling locations should be informed by fate and transport characteristics of the site type and source (see *Environmental Fate and Transport* fact sheet). Tools for determining the extent of established plumes may include transect surveys using direct push technology, followed by installation of monitoring wells, or other appropriate techniques such as high-resolution site characterization (USEPA 2016i). Potential secondary sources should be identified, for example, from irrigation or biosolids application, and other anthropogenic factors affecting fate and transport of PFAS-contaminated media.

Certain PFAS are present in ambient air, and may be elevated near sources such as landfills, WWTFs, fire training facilities, and manufacturing plants. Typical air sampling methods for PFAS include either glass fiber or quartz fiber filters and a sorbent material such as polymeric resin or polyurethane foam to collect both the particle and gas phases. Most

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methodologies in the literature collect the particle phase and then the gas phase; however, some studies developed a method to collect the gas phase first followed by the particle phase in efforts to not overestimate the particle phase concentration (Barber et al. 2007; Jahnke 2007b, 2009; Ahrens et al. 2011a, 2012).

2.2.3 Risk Assessment

Site-specific risk assessment is informed by data and information iteratively collected in the site characterization. Of the many PFAS that may be found at contaminated sites, the toxicity of PFOA and PFOS has been studied the most thoroughly. A substantial database of toxicity information is also available for some other PFAS including PFBA, PFBS, PFHxA, PFNA, and GenX, while there is limited publicly available information on toxicity of other PFAS that may be present at PFAS-contaminated sites. USEPA has established a Health Advisory for protection from a lifetime exposure to PFOA and PFOS from drinking water of 70 ppt for each compound individually, or the total of both. While many states use these USEPA Health Advisories as guidance for PFOA and PFOS, several states have developed more stringent levels for these compounds; some states have also developed standards or guidance for other PFAS of local concern (see the *Regulations, Guidance, and Advisories* fact sheet). Given that PFAS typically occur in complex mixtures, and human and environmental receptors are exposed to some PFAS-forming complex mixtures, evaluating the true risks at a site can be particularly challenging. In the absence of risk-based values for some of the PFAS that are detected and because additional PFAS not detected by the analytical method may be present, the investigation team should identify data gaps and communicate the impact that these gaps have on risk analyses. Data gaps and scientific uncertainty must be documented so that as site cleanup progresses and more information becomes available, the project team can reassess potential risks from the site and better communicate to the public how site decisions are made.

2.2.3.1 Human Receptors

The presence of PFAS in the environment and consumer product has resulted in detectable levels (most frequently PFOA, PFNA, PFOS and PFHxS) in the blood serum of most of the U.S. population (CDC 2017b). The total body burden of these PFAS results from exposure to the PFAS themselves and formation from precursors through metabolism in the body (Olsen et al. 2017; D'eon and Mabury 2011). Blood serum levels of these PFAS in the general population have generally decreased over time (CDC 2017a). Risk assessment of PFAS exposure for humans near contaminated sites must include both exposures prevalent in the general population, such as from the food supply and consumer products, and exposures from the contaminated site, such as drinking water, house dust, ambient air, and locally caught fish. Exposures from even relatively low levels (for example, below 70 ng/L) of long-chain PFAS in drinking water are much higher than total exposures in the general population not impacted by a contaminated site (Bartell 2017).

The tendency of some PFAS to bioaccumulate (ATSDR 2015a) is also a critical component in evaluating potential health effects; food chain routes of exposure should be considered. For example, PFOS and longer-chain perfluorinated sulfonates, and PFNA and longer-chain perfluorinated carboxylates, are known to bioaccumulate in fish, including in species used for food (Conder et al. 2008). Also, as a result of chronic ingestion of water and exposure to other materials containing PFAS, women may carry PFAS in their blood and breast milk. These PFAS are transferred to their baby during pregnancy and through breast feeding. Serum levels of long-chain PFAS rapidly increase in breast fed infants due to the PFAS levels present in breast milk and the higher fluid consumption rates of infants (Mogensen et al. 2015; Winkens et al. 2017; Fromme et al. 2010; Verner et al. 2016a, b).

2.2.3.2 Ecological Receptors

PFAS present a potential hazard to wildlife by direct and dietary exposure on both individual and population levels (Environment Canada 2006, 2012). Numerous studies have shown PFAAs, particularly PFSAs, are globally present in wildlife and may bioaccumulate in birds, fish, and mammals (including livestock); other animal classes are less studied (Houde et al. 2011; Lupton et al. 2014; OECD 2013). Biomagnification (in which concentrations increase with increasing trophic level) appears to be more complicated, occurring in some food webs but not others (Franklin 2016; Fang et al. 2014). Effects of PFAS exposure on wildlife vary widely by species and PFAS compound. Ecological toxicity information for many PFAS compounds is currently unavailable, while for others, data is limited and still evolving. Therefore, as site characterization activities for PFAS occur, the current state of the science should be reviewed before calculating ecological risk. More information is included in the *Environmental Fate and Transport* fact sheet.

3 Sampling

Sampling conducted to determine PFAS concentrations in water, soil, sediment, air, biota and other sources is similar to that for other chemical compounds, but with several additional specific considerations and protocols. If regulatory procedures, methods, or guidelines are inconsistent with the needs of a PFAS sampling program, then the governing

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agency should be contacted directly to determine an alternate approach or if an exception can be made. Other considerations for PFAS sampling include low laboratory detection limits, state and federal screening levels, and in some cases, cleanup criteria and potential for background concentrations of PFAS in the environment.

3.1 Equipment and Supplies

Many materials used in the course of environmental investigation can potentially contain PFAS. There is limited published research or guidance on how certain materials used by field staff affect sample results. Therefore, a conservative approach is recommended to exclude materials known to contain PFAS. Obtain and review all Safety Data Sheets (SDSs) before considering materials for use during PFAS sampling. Materials to avoid include:

- Teflon, polytetrafluoroethylene (PTFE)
- waterproof coatings containing PFAS
- food containers
- anything with fluoro in the name
- fluorinated ethylene propylene (FEP)
- ethylene tetrafluoroethylene (ETFE)
- low density polyethylene (LDPE), polyvinylidene fluoride (PVDF)

Many waterproof coatings contain PFAS, such as Gore-tex treated PPE or most waterproof papers, but some products are waterproofed with acceptable materials such as polyurethane, rubber, or PVC. Individual product specifications should be examined closely. In the case of Tyvek PPE, plain Tyvek does not contain PFAS while coated Tyvek does. In addition, materials incidentally transported to sites may contain PFAS. For example, fast food wrappers may contain PFAS. Due to the ubiquitous nature of PFAS, sampling crews must review all materials used to avoid contamination. Collection of quality assurance and quality control (QA/QC) samples is a useful tool to assess field contamination.

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Two guidance documents identify materials and equipment that can be used in PFAS-focused investigations, as well as materials that should be avoided because they are known or suspected to be potential sources of PFAS:

- *Bottle Selection and other Sampling Considerations When Sampling for Per-and Poly-Fluoroalkyl Substances (PFAS)* (USDOD EDQW 2017b)
- *Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoralkyl Substances (PFAS), Contaminated Sites Guidelines*, (Government of Western Australia, Department of Environment Regulation 2016)

Sometimes it is impossible to eliminate materials that affect PFAS results in samples. For example, these materials might be needed at sites where hazards warrant the use of specific personal protective equipment (PPE), where PFAS are the secondary or co-contaminant and the primary contaminant requires specific materials for proper sampling, or where the opportunity to collect a sample occurs before a proper sampling program is developed. When PFAS-containing equipment and supplies cannot be eliminated, increasing the equipment rinse blank samples will more thoroughly document the PFAS concentrations. In these situations, a thorough QA/QC program becomes even more important.

Not all PFAS are hydrophilic, and some are volatile. As a result, these chemicals may sorb to sampling equipment and supplies or be lost from samples during sample collection. Preliminary data suggest that sorption may occur quickly. Additionally, volatile losses have not yet been characterized. Until they are better quantified, sampling efforts should consider whether these losses would affect project objectives and adjust accordingly.

3.2 Bottle Selection and Sample Amount

Containers should be specified in the analytical method, provided by the laboratory selected to perform the analyses, and should be certified by the laboratory to be PFAS-free. The term *PFAS-free* is a method or project-defined concentration level (for example, < 1/2 the limit of quantitation for the specific compound of interest). USEPA Method 537, Version 1.1 (September 2009) requires the use of 250 mL polypropylene containers and caps/lids for drinking water sampling (Shoemaker, Grimmett, and Boutin 2009). Currently, USEPA has not issued guidance or analytical methods for any sample media other than drinking water. Depending on the analytical method used or program (for example state or DOD) requirements, polypropylene or high-density polyethylene (HDPE) bottles with unlined plastic caps are typically used (USDOD EDQW 2017b).

Best practices in sample preparation must be used when selecting the size, volume, and representativeness of samples. To minimize effects from analyte sorption on sample containers, the laboratory must analyze the entire sample, including the sample container rinsate. The project screening or applicable regulatory levels, and the expected or potential concentration of the analytes, are also relevant. If the sample is known to contain high concentrations of PFAS (for example, AFFF formulations), loss is negligible and therefore the entire sample does not need to be used.

Because the concentration level of PFAS in aqueous samples determines whether the whole sample or an aliquot is used in the laboratory preparation, the sampler should collect an additional volume of each sample in a separate container. Then, the laboratory can screen the extra sample for high concentrations without affecting the final sample result. For soil or sediment, obtaining a representative subsample in the laboratory is critical, so the entire sample should be homogenized in the laboratory prior to subsampling. Coordinating with the laboratory is crucial to determine the appropriate sample container volumes for environmental media other than drinking water.

3.3 Sample Preservation, Shipping, Storage, and Hold Times

USEPA Method 537, Version 1.1 contains specific requirements for drinking water sample preservation, shipping, storage, and holding times (Shoemaker, Grimmett, and Boutin 2009). Currently, there is no USEPA guidance or requirement for other sample media. The chemical preservation required by Method 537, Trizma, is added for buffering and free chlorine removal and applicable to DW samples only. Until additional information is available, the thermal preservation, shipping, storage, and holding times contained in USEPA Method 537, Version 1.1 should be used for all other sample media except biota. For biota samples (for example, vegetation, fish), the samples should be frozen to limit microbial growth until sample preparation is performed at the laboratory. Microbial growth may result in PFAAs values biased high due to biodegradation of precursor compounds; however, these effects have not been well studied.

3.4 Decontamination Procedures

Field sampling equipment, including oil/water interface meters, water level indicators, and other nondedicated equipment used at each sample location, require cleaning between use. The SDSs of detergents or soaps used in decontamination procedures should be reviewed to ensure fluoro-surfactants are not listed as ingredients. Use laboratory-certified PFAS-free water for the final rinse during decontamination of sampling equipment. Decontaminate larger equipment (for example, drill rigs and large downhole drilling and sampling equipment) with potable water using a high-pressure washer or steam. To the extent practical, rinse parts of equipment coming in direct contact with samples with PFAS-free water. Heavy equipment is best cleaned within a decontamination facility or other means of containment (for example, a bermed, lined pad and sump, or a portable, self-contained decontamination booth). Potable water sources should be analyzed in advance for PFAS. Wherever possible, rinse equipment with PFAS-free water immediately before use.

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3.5 Field QC

Field quality control (QC) samples are a means of assessing quality from the point of collection. Such QC samples include, but are not limited to, field reagent blanks, equipment rinse blanks, and sample duplicates. USEPA Method 537, Version 1.1 contains specific requirements for the QC samples that must accompany drinking water samples. Collection and analysis of QC samples are important for PFAS analyses because of very low detection limits and widespread commercial use (historical and current) of PFAS containing products.

3.6 Sampling Precautions

Standard sampling procedures can be used at most PFAS sites. However, there may be some exceptions and additional considerations related to PFAS behavior, and issues associated with potential use of PFAS-containing or adsorbing sampling equipment and supplies.

3.6.1 Groundwater

The most inert material (for example, stainless steel, silicone, and HDPE), with respect to known or anticipated contaminants in wells should be used whenever possible. Dedicated sampling equipment installed in existing wells prior to investigation should be thoroughly checked to ensure that the equipment is PFAS-free. For long-term investigations, samples may be collected in duplicate with and without existing dedicated equipment. If PFAS analyses show that the equipment does not affect results, the equipment may be kept and used long term. This determination depends on project-specific requirements, however, and should only be used by a project team with full disclosure to all stakeholders.

3.6.2 Surface Water

To avoid cross-contamination from sampling materials to sample media, the outside of all capped sample containers should be rinsed multiple times with the surface water being sampled before filling the containers. When site conditions require, remote sampling into sample containers can be accomplished by clamping the container onto the end of a clean extension rod. The extension rod must be made of PFAS-free material and have been decontaminated. Within the context of sample collection objectives, the sample location in the water column should consider the potential stratification of PFAS in solution and their tendency to accumulate at the air/water interface. For more information on stratification, see the *Environmental Fate and Transport* fact sheet.

3.6.3 Porewater

Peristaltic pumps with silicone and HDPE tubing are typically used for porewater sample collection, along with push point samplers, porewater observation devices (PODs), or drive point piezometers. Push point samples and drive point piezometers are made of stainless steel, while PODs consist of slotted PVC pipe and silicone tubing. These samplers should be dedicated and not reused across a site or multiple sites.

3.6.4 Soil/Sediment

Most core and grab sampling devices are constructed of stainless steel. Some core samplers include an HDPE sleeve inserted in the core barrel to retain the sample. PPE such as waders and personal flotation devices may be required. Ensure that materials that contact the media to be sampled do not have water-resistant coatings which contain PFAS.

3.6.5 Fish

The species of fish collected, as well as the portion of fish sampled (whole versus fillet), depends on the project goals (for example, ecological risk or human health). Studies have shown the majority of the PFAS in fish are stored in the organs, not the flesh (Martin et al. 2004; Yamada et al. 2014). Communicating project objectives to the laboratory is important prior to field work in order to determine the necessary quantity and quality of tissue, fish handling requirements, laboratory sample preparation (including single fish or composite fish samples, and whole or fillet preparation), and packing and shipping requirements.

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3.6.6 Potential high concentration samples

The CSM or previous sampling may indicate areas of high concentrations of PFAS for which single-use, disposable equipment is recommended. If single-use is not possible, take additional precautions such as implementing a greater frequency of decontamination blanks and not reusing equipment to sample potentially low PFAS concentration samples. High concentration samples should be segregated during shipping to the laboratory.

Some projects may require the analysis of AFFF product that has been used at the site. All AFFF product samples must be considered high concentration samples. These samples should be segregated from other samples during sampling and shipping to avoid cross contamination. Samples that may contain high concentrations of PFAS should be clearly identified on the *Sample Chain of Custody* that is shipped with the samples. Field test kits are available for PFAS but have not been fully evaluated. While these kits cannot achieve low detection limits, they could be helpful in screening for potential high concentrations of PFAS in the field.

4 Quantitative Analysis

USEPA Method 537, Version 1.1 contains specific requirements for sample preparation and analysis of drinking water samples. Currently, there are no USEPA methods for the preparation and analysis of other sample media. However, other published methods may apply:

- ISO Method 25101 (ISO 2009)
- ASTM D7979 (ASTM 2017b)
- ASTM D7968 (ASTM 2017a)

To evaluate the laboratory's ability to meet the needs of a project, the laboratory's analytical procedure should be reviewed as part of the laboratory selection process. In addition, performance data such as concentrations observed in lab blanks and matrix spike recovery are necessary.

4.1 Sample Preparation

The sample preparation procedure should be specified in the sample analysis procedure and should be included as part of the sample and analysis plan (SAP) or quality assurance project plan (QAPP). This procedure should demonstrate that extreme care is taken to prevent sample contamination during preparation and extraction. All supplies must be checked and confirmed as PFAS-free prior to sample preparation. Intermittent contamination can occur due to vendor supply or manufacturing changes; therefore, each lot of supplies should be verified and documented prior to use.

Because sample preparation may vary in different analytical procedures, the laboratory should document its preparation process for the samples. A critical step in the laboratory's preparation process is ensuring a representative sample or subsample is used for analysis. For all media, sample transfers should be minimized. Sample filtration to eliminate solid particulate from aqueous samples is not recommended because PFAS losses can occur due to adsorption of PFAS onto filters.

The entire aqueous sample received should be prepared and the sample container appropriately rinsed. Aqueous samples that are prepared using the whole sample must be extracted using SPE. The exception to this practice is samples containing high concentrations of PFAS, because each type of solid phase extraction cartridge has a defined capacity to retain PFAS analytes. Exceeding this capacity results in a low bias in PFAS results. In these instances, to prevent this bias, samples can be prepared using serial dilution techniques or analyzed using direct injection (for example, ASTM D7979). Most laboratories screen samples using a small volume sample to determine if it contains PFAS at concentrations too high for SPE sample preparation and analysis. For solid samples, the laboratory homogenizes the sample before subsampling and extraction.

To account for biases resulting from preparation steps, internal standards should be added to all samples (preferably extracted internal standards that are isotopically-labeled analogs of each analyte, if commercially available). The addition of internal standards to the sample should be clearly documented. Internal standards should be added to the sample at different steps in the process, depending on the sample preparation process used. Internal standards should also be added to whole field samples in the field container (SPE extraction samples) after subsampling, prior to addition of extraction solvent for soil or sediment samples, and after final dilution for serial dilution prepared samples (USDOD 2017a).

Depending on the analytical method used, cleanup procedures (for example, graphitized carbon) may be used on samples when matrix interferences (for example, bile salts and gasoline range organics) could be present. ENVI-Carb cleanup removes cholic acids, a known interference in fish tissue sample. The procedure should clearly state what type of cleanup process is used and in what instances.

The analytical procedure should describe what batch QC samples are prepared with each media type. Batch QC samples might include method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD), sample duplicate (SD), matrix spike (MS), and matrix spike duplicate (MSD). Additional QC may also be included. For samples with high concentrations of PFAS, in addition to an MS and an MSD, an LCSD and an SD may be warranted. The SD should be prepared using a different aliquot from the same sample bottle to create a second set of serial dilutions. Review of the laboratory's procedure should ensure that the laboratory is capable of using the batch QC needed for the project, including meeting the project's QC acceptance criteria.

4.2 Sample Analysis

Currently, the analytical detection method of choice for PFAS analysis is liquid chromatography-mass spectrometry-mass spectrometry (LC/MS/MS), which is especially suited for analysis of ionic compounds, such as the PFSAs and PFCAs. Gas chromatography-mass spectrometry (GC/MS) can also be used for PFAS analysis, specifically the neutral and nonionic analytes, such as the fluorotelomer alcohols (FTOHs), perfluoroalkane sulfonamides, and perfluoroalkane sulfonamido ethanols. Currently, LC/MS/MS analysis of PFAS is widely available, whereas GC/MS analysis has limited commercial availability.

LC/MS/MS methods developed by laboratories may be based on USEPA Method 537, Version 1.1. The USEPA method does not contain steps to alleviate matrix interference issues potentially found in other sample media and does not contain steps to prepare solid sample media. Methods for other sample media may include extraction or sample preparation procedures for other matrices, use of isotope dilution, the addition of other PFAS analytes, and confirmation using confirmatory ions and ion ratios. Because these modifications are not standardized, analytical methods can result in greatly varied data, precision, and accuracy. Laboratories should provide performance data for the relevant media

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for each project. The USDOD EDQW has attempted to standardize many of these modifications through requirements contained in the USDOD Environmental Laboratory Accreditation Program (USDOD ELAP) document, the DOD *Quality Systems Manual for Environmental Laboratories* (DOD QSM), Version 5.1, Appendix B, Table B-15 (USDOD 2017a).

Certified analytical standards are available from several manufacturers. Products may have variable purity and isomer profiles, which may compromise the accuracy, precision, and reproducibility of data. Only certified standards of the highest purity available, for example, American Chemical Society grade, can be used for accurate quantitation. Standards containing linear and branched isomers are not commercially available for all applicable analytes. Currently, such standards are only available for PFOS and perfluorohexane sulfonic acid (PFHxS). Technical grades which contain branched and linear isomers are available for other PFAS, but these standards do not have the accuracy needed for quantitation purposes. These standards may, however, be qualitatively useful for verifying which peaks represent the branched isomers. Methods should specify the isomers quantified as well as the isomers included in standards used for quantitation purposes.

Isotope dilution is a quantitation technique that considers sample matrix effects on each individual PFAS quantitation in the most precise manner possible. This technique quantifies analytes of interest against the isotopically labeled analogs of the analytes, which are added to the sample prior to and after sample preparation. Addition prior to preparation helps account for loss of analyte during the preparation process, while addition after preparation to an aliquot of the sample extract accounts for the bias associated with the instrumentation. Methods using isotope dilution should include isotope recovery for each sample and analyte in data reports. Isotope analog recoveries should be reported, and minimum/maximum isotope recoveries may be required by specific analytical procedures. Low isotope recovery may indicate that quantitation was inadequate; the data are then reported as estimated values.

Mass calibration should occur at the frequency recommended by the instrument manufacturer and as needed based on QC indicators, such as calibration verifications. The instrument blanks, calibration curve, and initial and continual calibration verification requirements should be consistent with those published for other LC/MS/MS methods. The lowest calibration point should be a concentration at or below the limit of quantitation. A standard at the limit of quantitation concentration should be analyzed with each analytical batch to document the instrument's ability to accurately quantitate down to that concentration. Instrument blanks are critical in determining if the instrument is potentially affecting PFAS concentrations in samples.

Quantification by LC/MS/MS may be accomplished using a variety of techniques. For relatively simple matrices such as drinking water, Method 537 quantifies analytes by comparing the product ion of one precursor ion and retention time in samples to calibration standards. For more complex matrices, additional product ions and their ion ratios can be used to distinguish analytes from matrix interference. In an MS/MS system, an analyte can be fractured into more than one ion. By monitoring the area of each ion and comparing the ratio of those area counts, a more definitive identification can be made. This identification allows the analyst to distinguish true target analytes from false positives. This more detailed quantification is not required for drinking water matrices, but it is useful for more complex matrices.

As part of the laboratory selection process, the laboratory's analytical procedure should be evaluated to ensure these parameters are addressed in the documentation provided. In addition, the acceptance criteria for all the analytical QC elements should be evaluated to ensure that they are set at levels that meet the project's measurement quality objectives (MQOs). For DOD projects, these criteria can be found in the DOD QSM, Version 5.1, Appendix B, Table B-15 (USDOD 2017a).

4.3 Data Evaluation

Data evaluation is a critical step in any project; however, it becomes even more important when nonstandard methods are used, such as for PFAS. Without a standard method for media other than drinking water, laboratories' methods may vary greatly in their precision and accuracy. Over time, these methods become optimized based on new knowledge about sampling and analytical biases. Advances in instrumentation and analytical supplies (such as standards availability and improved analytical columns) often occur as well because of commercial demand. As a result, the precision and accuracy of the data generated by laboratories can change significantly over time, making it difficult to compare data generated over an extended time period. Thus, data evaluation should be performed using the most current knowledge on the state of science of PFAS.

Precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) parameters should be assessed because they guide data evaluation (field collection and laboratory information). Data are reviewed in a

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systematic way by looking at the results of each QC indicator of the PARCCS parameters (for example, spike recoveries and method blanks) to obtain an understanding of the overall quality of the data. The most important goal of data evaluation is to ensure that any limitations to the PFAS data generated are understood, which establishes confidence that the data meet site-specific needs. More information is available in the IDQTF (2005) and USEPA (2000a) Quality Assurance Project Plan documents.

5 Qualitative Analysis

Several methods employing indirect measurement have been developed that more comprehensively assess the range of PFAS contamination at a site. Two techniques are available to measure organofluorine (Dauchy et al. 2017; Willach, Brauch, and Lange 2016; Ritter et al. 2017):

- Adsorbable organic fluorine (AOF) paired with combustion ion chromatography (CIC) measure the combusted organofluorine content of a sample as fluoride on an IC.
- Proton induced gamma-ray emission (PIGE) spectroscopy measures elemental fluorine isolated on a thin surface.

Both techniques isolate organofluorine material on a sorptive material such as activated carbon or an anion exchange cartridge prior to measurement; neither technique is currently commercially available. A third technique, total oxidizable precursor assay (TOP assay or TOPA) converts PFAA precursor compounds to PFAAs through an oxidative digestion. The increase in PFAAs measured after the TOP assay, relative to before, is a conservative estimate of the total concentration of PFAA precursors present in a sample, because not all PFAS present will be subject to quantitation or reaction, and will remain as undetected PFAS. The PFAAs generated have perfluoroalkyl chain lengths equal to, or shorter than, the perfluoroalkyl chain lengths present in the precursors (Houtz et al. 2013; Houtz and Sedlak 2012; Weber et al. 2017; Dauchy et al. 2017). Finally, quantitative time of flight mass spectrometry (QTOF-MS) can be used to determine both the chemical formula and structure of unknown PFAS in a sample, but analytical standards are required for unequivocal structural identification.

Library research, preliminary identification of potential PFAS sources, and information gathered from patents can assist in the identification of PFAS using QTOF-MS (Newton et al. 2017; Moschet et al. 2017; Barzen-Hanson et al. 2017). These methods are not standardized through a published USEPA method and range in commercial availability. To date, these methods have not undergone multilaboratory validation. As a result, TOP assay, the most widely commercially available of the techniques, is typically accepted as a means of determining PFAS load on remediation substances to estimate the replacement cycle, but not for site characterization.

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6 References and Acronyms

The references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list that is available on the ITRC web site. The combined acronyms list is also available on the ITRC web site.

Per- and Polyfluoroalkyl Substances (PFAS) Team Contacts

Robert Mueller • New Jersey Department of Environmental Protection
609-984-3910 • Bob.Mueller@dep.nj.gov

Virginia Yingling • Minnesota Department of Health
651-343-2890 • virginia.yingling@state.mn.us

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EPA 537 (PFAS) Field Sampling Guidelines

PLEASE READ INSTRUCTIONS ENTIRELY PRIOR TO SAMPLING EVENT

Sampling for PFAS via EPA 537 can be challenging due to the prevalence of these compounds in consumer products. The following guidelines are strongly recommended when conducting sampling.

Reference-NHDES https://www.des.nh.gov/organization/divisions/waste/hwrb/documents/pfc-stakeholder-notification-20161122.pdf

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FIELD CLOTHING and PPE

- No clothing or boots containing Gore-Tex®
- All safety boots made from polyurethane and PVC
- No materials containing Tyvek®
- Do not use fabric softener on clothing to be worn in field
- Do not used cosmetics, moisturizers, hand cream, or other related products the morning of sampling
- Do not use unauthorized sunscreen or insect repellant (see reference above for acceptable products)

FOOD CONSIDERATIONS

No food or drink on-site with exception of bottled water and/or hydration drinks (i.e., Gatorade and Powerade) that is available for consumption only in the staging area

OTHER RECOMMENDATIONS

Sample for PFAS first! Other containers for other methods may have PFAS present on their sampling containers

SAMPLE CONTAINERS

- All sample containers made of HDPE or polypropylene
- Caps are unlined and made of HDPE or polypropylene (no Teflon® -lined caps)

WET WEATHER (AS APPLICABLE)

Wet weather gear made of polyurethane and PVC only

EQUIPMENT DECONTAMINATION

- "PFAS-free" water on-site for decontamination of sample equipment. No other water sources to be used
- Only Alconox and Liquinox can be used as decontamination materials

FIELD EQUIPMENT

- Must not contain Teflon® (aka PTFE) or LDPE materials
- All sampling materials must be made from stainless steel, HDPE, acetate, silicon, or polypropylene
- No waterproof field books can be used
- No plastic clipboards, binders, or spiral hard cover notebooks can be used
- No adhesives (i.e. Post-It® Notes) can be used
- Sharpies and permanent markers not allowed; regular ball point pens are acceptable
- Aluminum foil must not be used
- Keep PFC samples in separate cooler, away from sampling containers that may contain PFAS
- Coolers filled with regular ice only - Do not use chemical (blue) ice packs

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EPA 537 (PFAS) Field Sampling Guidelines

PLEASE READ INSTRUCTIONS ENTIRELY PRIOR TO SAMPLING EVENT

Sampler must wash hands before wearing nitrile gloves in order to limit contamination during sampling. Each sample set requires a set of containers to comply with the method as indicated below. **Sample set is composed of samples collected from the same sample site and at the same time.*

Container Count	Container Type	Preservative
3 Sampling Containers - Empty	250 mL container	Pre preserved with 1.25 g Trizma
1 Reagent Water for Field Blank use	250 mL container	Pre preserved with 1.25 g Trizma
P1 Field Blank (FRB) - Empty	250 mL container	Unpreserved

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Sampling container must be filled to the neck. For instructional purposes a black line has been drawn to illustrate the required fill level for each of the 3 Sample containers

Field blanks are recommended and the containers have been provided, please follow the instructions below.

Field Blank Instructions:

1. Locate the Reagent Water container from the bottle order. The Reagent Water container will be pre-filled with PFAS-free water and is preserved with Trizma.
2. Locate the empty container labeled "Field Blank".
3. Open both containers and proceed to transfer contents of the "Reagent Water" container into the "Field Blank" container.
4. If field blanks are to be analyzed, they need to be noted on COC, and will be billed accordingly as a sample.

Both the empty Reagent Water container and the filled Field Blank container must be returned to the lab along with the samples taken.

Sampling Instructions:

1. Each sampling event requires 3 containers to be filled to the neck of the provided containers for each sampling location.
2. Before sampling, remove faucet aerator, run water for 5 min, slow water to flow of pencil to avoid splashing and fill sample containers to neck of container (as previously illustrated) and invert 5 times.
3. Do not overfill or rinse the container.
4. Close containers securely. Place containers in sealed ZipLoc® bags, and in a separate cooler (no other container types).
5. Ensure Chain-of-Custody and all labels on containers contain required information. Place sample, Field Blank and empty Reagent Blank containers in ice filled cooler (do not use blue ice) and return to the laboratory. Samples should be kept at 4°C ±2. Samples must not exceed 10°C during first 48 hours after collection. Hold time is 14 days.

Please contact your Alpha Analytical project manager with additional questions or concerns.

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ATTACHMENT E

LABORATORY INFORMATION

DRAFT

THE STATE of ALASKA
GOVERNOR BILL WALKER

Department of Environmental Conservation

DIVISION OF SPILL PREVENTION AND RESPONSE
Contaminated Sites Program
Laboratory Approval Program

555 Cordova Street
Anchorage, Alaska 99501
Main: 907.465.5390
Fax: 907.269.7649
cs.lab.cert@alaska.gov

February 6, 2018

Crystal Pollock
TestAmerica – Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

RE: Contaminated Sites Laboratory Approval **17-020**

Dear Ms. Pollock,

Thank you for submitting an application to the Alaska Department of Environmental Conservation's Contaminated Sites Laboratory Approval Program (CS-LAP), on October 31, 2017. Based on your lab's National Environmental Laboratory Accreditation Program (NELAP) approval through the Oregon Environmental Laboratory Accreditation Program (ORELAP), and Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) approval through the ANSI-ASQ National Accreditation Board (ANAB), TestAmerica – Sacramento, located at the above address, is granted ***Approved*** status to perform the analyses listed in the attached *Scope of Approval*, for Alaska contaminated sites projects, including underground storage tanks and leaking underground storage tank sites (UST/LUST), under the July 1, 2017 amendments to 18 AAC 78. This approval expires on ***January 20, 2021***.

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Be aware that **any** changes in your NELAP or DoD-ELAP approval status must be reported to the CS program within 3 business days. Failure to do so will result in revocation of **all** CS-LAP approvals for a period of one year. Notification should be in writing sent to cs.lab.cert@alaska.gov. We recommend also contacting the CS-LAP by telephone to verify that the message was received.

To report any changes in your lab's contact information (i.e. lab director, business name, location, etc.), please complete the form found at http://dec.alaska.gov/spar/csp/LabApproval/ApplyForApproval.htm and submit to cs.submittals@alaska.gov.

To apply for renewal of your approval, please complete the application found at http://dec.alaska.gov/spar/csp/LabApproval/ApplyForApproval.htm and submit to cs.submittals@alaska.gov. The required documentation must be submitted for renewal no later than 30 days before your date of expiration.

Please remember to include the laboratory's ID number, listed above, on all correspondence concerning the laboratory.

If you have any questions, please contact the CS-LAP at (907) 465-5390, or by email at cs.lab.cert@alaska.gov. Labs are also highly encouraged to join the CS-LAP listserv by going to http://list.state.ak.us/mailman/listinfo/cs.lab.approval.

Respectfully,

Brian Englund
Alaska CS Lab Approval Officer

Attachment: Scope of Approval

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Acenaphthene	83-32-9	8270C	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270C-SIM	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270D	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270D-SIM	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	TO-13A SIM	---	---	**X**	ANAB
Acenaphthylene	208-96-8	8270C	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270C-SIM	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270D	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270D-SIM	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	TO-13A SIM	---	---	**X**	ANAB
Acetone	67-64-1	8260B	**X**	**X**	---	ANAB
Acetone	67-64-1	8260C	**X**	**X**	---	ANAB
Acetone	67-64-1	TO-15	---	---	**X**	ANAB
Aldrin	309-00-2	8081A	**X**	**X**	---	ANAB
Aldrin	309-00-2	8081B	**X**	**X**	---	ANAB
Aldrin	309-00-2	TO-10A	---	---	**X**	ORELAP
Ammonium Perchlorate	7790-98-9		**X**	**X**	---	ANAB
Anthracene	120-12-7	8270C	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270C-SIM	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270D	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270D-SIM	**X**	**X**	---	ANAB
Anthracene	120-12-7	TO-13A SIM	---	---	**X**	ANAB
Antimony (metallic)	7440-36-0	6010B	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6010C	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6020A	**X**	**X**	---	ANAB
Aroclor 1242	53469-21-9	TO-10A	---	---	**X**	ANAB
Aroclor 1254	11097-69-1	TO-10A	---	---	**X**	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Aroclor 1260	11096-82-5	TO-10A	---	---	**X**	ANAB
Arsenic, Inorganic	7440-38-2	6010B	**X**	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6010C	**X**	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6020A	**X**	**X**	---	ANAB
Barium	7440-39-3	6010B	**X**	**X**	---	ANAB
Barium	7440-39-3	6010C	**X**	**X**	---	ANAB
Barium	7440-39-3	6020A	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270C	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270C-SIM	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270D	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270D-SIM	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	TO-13A SIM	---	---	**X**	ANAB
Benzaldehyde	100-52-7	8270C	**X**	**X**	---	ANAB
Benzaldehyde	100-52-7	8270D	**X**	**X**	---	ANAB
Benzene	71-43-2	8260B	**X**	**X**	---	ANAB
Benzene	71-43-2	8260C	**X**	**X**	---	ANAB
Benzene	71-43-2	TO-15	---	---	**X**	ANAB
Benzene	71-43-2	TO-15 SIM	---	---	**X**	ANAB
Benzo[a]pyrene	50-32-8	8270C	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270C-SIM	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270D	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270D-SIM	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	TO-13A SIM	---	---	**X**	ANAB
Benzo[b]fluoranthene	205-99-2	8270C	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270C-SIM	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270D	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270D-SIM	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Benzo[b]fluoranthene	205-99-2	TO-13A SIM	---	---	**X**	ANAB
Benzo[g,h,i]perylene	191-24-2	8270C	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270C-SIM	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270D	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270D-SIM	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	TO-13A SIM	---	---	**X**	ANAB
Benzo[k]fluoranthene	207-08-9	8270C	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270C-SIM	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270D	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270D-SIM	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	TO-13A SIM	---	---	**X**	ANAB
Benzoic Acid	65-85-0	8270C	**X**	**X**	---	ANAB
Benzoic Acid	65-85-0	8270D	**X**	**X**	---	ANAB
Benzyl Alcohol	100-51-6	8270C	**X**	**X**	---	ANAB
Benzyl Alcohol	100-51-6	8270D	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6010B	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6010C	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6020A	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270C	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270D	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270D-SIM	**X**	**X**	---	ANAB
Bis(2-ethylhexyl)phthalate (DEHP)	117-81-7	8270C	**X**	**X**	---	ANAB
Bis(2-ethylhexyl)phthalate (DEHP)	117-81-7	8270D	**X**	**X**	---	ANAB
Bromobenzene	108-86-1	8260B	**X**	**X**	---	ANAB
Bromobenzene	108-86-1	8260C	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260B	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260C	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Bromodichloromethane	75-27-4	TO-15	---	---	**X**	ANAB
Bromodichloromethane	75-27-4	TO-15 SIM	---	---	**X**	ANAB
Bromoform	75-25-2	8260B	**X**	**X**	---	ANAB
Bromoform	75-25-2	8260C	**X**	**X**	---	ANAB
Bromoform	75-25-2	TO-15	---	---	**X**	ANAB
Bromomethane	74-83-9	8260B	**X**	**X**	---	ANAB
Bromomethane	74-83-9	8260C	**X**	**X**	---	ANAB
Bromomethane	74-83-9	TO-15	---	---	**X**	ANAB
Butadiene, 1,3-	106-99-0	8260C-SIM	**X**	**X**	---	ANAB
Butadiene, 1,3-	106-99-0	TO-15	---	---	**X**	ANAB
Butadiene, 1,3-	106-99-0	TO-15 SIM	---	---	**X**	ANAB
Butyl Benzyl Phthalate	85-68-7	8270C	**X**	**X**	---	ANAB
Butyl Benzyl Phthalate	85-68-7	8270D	**X**	**X**	---	ANAB
Butylbenzene, n-	104-51-8	8260B	**X**	**X**	---	ANAB
Butylbenzene, n-	104-51-8	8260C	**X**	**X**	---	ANAB
Butylbenzene, sec-	135-98-8	8260B	**X**	**X**	---	ANAB
Butylbenzene, sec-	135-98-8	8260C	**X**	**X**	---	ANAB
Butylbenzene, tert-	98-06-6	8260B	**X**	**X**	---	ANAB
Butylbenzene, tert-	98-06-6	8260C	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6010B	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6010C	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6020A	**X**	**X**	---	ANAB
Carbon Disulfide	75-15-0	8260B	**X**	**X**	---	ANAB
Carbon Disulfide	75-15-0	8260C	**X**	**X**	---	ANAB
Carbon Disulfide	75-15-0	TO-15	---	---	**X**	ANAB
Carbon Tetrachloride	56-23-5	8260B	**X**	**X**	---	ANAB
Carbon Tetrachloride	56-23-5	8260C	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Carbon Tetrachloride	56-23-5	TO-15	---	---	**X**	ANAB
Carbon Tetrachloride	56-23-5	TO-15 SIM	---	---	**X**	ANAB
Chlordane, Total	12789-03-6	8081A	**X**	**X**	---	ANAB
Chlordane, Total	12789-03-6	8081B	**X**	**X**	---	ANAB
Chlordane, Total	12789-03-6	TO-10A	---	---	**X**	ORELAP
Chlordane, α-	5103-71-9	8081A	**X**	**X**	---	ANAB
Chlordane, α-	5103-71-9	8081B	**X**	**X**	---	ANAB
Chlordane, γ-	5103-74-2	8081A	**X**	**X**	---	ANAB
Chlordane, γ-	5103-74-2	8081B	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270C	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270D	**X**	**X**	---	ANAB
Chlorobenzene	108-90-7	8260B	**X**	**X**	---	ANAB
Chlorobenzene	108-90-7	8260C	**X**	**X**	---	ANAB
Chlorobenzene	108-90-7	TO-15	---	---	**X**	ANAB
Chlorobenzene	108-90-7	TO-15 SIM	---	---	**X**	ANAB
Chloroform	67-66-3	8260B	**X**	**X**	---	ANAB
Chloroform	67-66-3	8260C	**X**	**X**	---	ANAB
Chloroform	67-66-3	TO-15	---	---	**X**	ANAB
Chloroform	67-66-3	TO-15 SIM	---	---	**X**	ANAB
Chloromethane	74-87-3	8260B	**X**	**X**	---	ANAB
Chloromethane	74-87-3	8260C	**X**	**X**	---	ANAB
Chloromethane	74-87-3	TO-15	---	---	**X**	ANAB
Chloromethane	74-87-3	TO-15 SIM	---	---	**X**	ANAB
Chloronaphthalene, Beta-	91-58-7	8270C	**X**	**X**	---	ANAB
Chloronaphthalene, Beta-	91-58-7	8270D	**X**	**X**	---	ANAB
Chlorophenol, 2-	95-57-8	8270C	**X**	**X**	---	ANAB
Chlorophenol, 2-	95-57-8	8270D	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Chromium (Total)	7440-47-3	6010B	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	6010C	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	6020A	**X**	**X**	---	ANAB
Chromium (VI)	18540-29-9	7196	---	**X**	---	ANAB
Chrysene	218-01-9	8270C	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270C-SIM	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270D	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270D-SIM	**X**	**X**	---	ANAB
Chrysene	218-01-9	TO-13A SIM	---	---	**X**	ANAB
Copper	7440-50-8	6010B	**X**	**X**	---	ANAB
Copper	7440-50-8	6010C	**X**	**X**	---	ANAB
Copper	7440-50-8	6020A	**X**	**X**	---	ANAB
Cresol, m- (3-Methylphenol)	108-39-4	8270C	**X**	**X**	---	ANAB
Cresol, m- (3-Methylphenol)	108-39-4	8270D	**X**	**X**	---	ANAB
Cresol, o- (2-Methylphenol)	95-48-7	8270C	**X**	**X**	---	ANAB
Cresol, o- (2-Methylphenol)	95-48-7	8270D	**X**	**X**	---	ANAB
Cresol, p- (4-Methylphenol)	106-44-5	8270C	**X**	**X**	---	ANAB
Cresol, p- (4-Methylphenol)	106-44-5	8270D	**X**	**X**	---	ANAB
Cumene (Isopropylbenzene)	98-82-8	8260B	**X**	**X**	---	ANAB
Cumene (Isopropylbenzene)	98-82-8	8260C	**X**	**X**	---	ANAB
Cyclohexane	110-82-7	8260B	**X**	**X**	---	ANAB
Cyclohexane	110-82-7	8260C	**X**	**X**	---	ANAB
Cyclohexane	110-82-7	TO-15	---	---	**X**	ANAB
DDD, 4,4'-	72-54-8	8081A	**X**	**X**	---	ANAB
DDD, 4,4'-	72-54-8	8081B	**X**	**X**	---	ANAB
DDE, 4,4'-	72-55-9	8081A	**X**	**X**	---	ANAB
DDE, 4,4'-	72-55-9	8081B	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
DDE, 4,4'-	72-55-9	TO-10A	---	---	**X**	ORELAP
DDT, 4,4'-	50-29-3	8081A	**X**	**X**	---	ANAB
DDT, 4,4'-	50-29-3	8081B	**X**	**X**	---	ANAB
DDT, 4,4'-	50-29-3	TO-10A	---	---	**X**	ORELAP
Dibenz[a,h]anthracene	53-70-3	8270C	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270C-SIM	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270D	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270D-SIM	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	TO-13A SIM	---	---	**X**	ANAB
Dibenzofuran	132-64-9	8270C	**X**	**X**	---	ANAB
Dibenzofuran	132-64-9	8270D	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260B	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260C	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	TO-15	---	---	**X**	ANAB
Dibromochloromethane	124-48-1	TO-15 SIM	---	---	**X**	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260B	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260C	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	TO-15	---	---	**X**	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260B	**X**	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260C	**X**	**X**	---	ANAB
Dibutyl Phthalate	84-74-2	8270C	**X**	**X**	---	ANAB
Dibutyl Phthalate	84-74-2	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8260B	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8260C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	TO-15	---	---	**X**	ANAB

DRAFT

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dichlorobenzene, 1,2-	95-50-1	TO-15 SIM	---	---	**X**	ANAB
Dichlorobenzene, 1,3-	541-73-1	8260B	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	8260C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	TO-15	---	---	**X**	ANAB
Dichlorobenzene, 1,3-	541-73-1	TO-15 SIM	---	---	**X**	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260B	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	TO-15	---	---	**X**	ANAB
Dichlorobenzene, 1,4-	106-46-7	TO-15 SIM	---	---	**X**	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270D	**X**	**X**	---	ANAB
Dichlorodifluoromethane	75-71-8	8260B	**X**	**X**	---	ANAB
Dichlorodifluoromethane	75-71-8	8260C	**X**	**X**	---	ANAB
Dichlorodifluoromethane	75-71-8	TO-15	---	---	**X**	ANAB
Dichlorodifluoromethane	75-71-8	TO-15 SIM	---	---	**X**	ANAB
Dichloroethane, 1,1-	75-34-3	8260B	**X**	**X**	---	ANAB
Dichloroethane, 1,1-	75-34-3	8260C	**X**	**X**	---	ANAB
Dichloroethane, 1,1-	75-34-3	TO-15	---	---	**X**	ANAB
Dichloroethane, 1,1-	75-34-3	TO-15 SIM	---	---	**X**	ANAB
Dichloroethane, 1,2-	107-06-2	8260B	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	8260C	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	TO-15	---	---	**X**	ANAB
Dichloroethane, 1,2-	107-06-2	TO-15 SIM	---	---	**X**	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dichloroethylene, 1,1-	75-35-4	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	8260C	**X**	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	TO-15	---	---	**X**	ANAB
Dichloroethylene, 1,1-	75-35-4	TO-15 SIM	---	---	**X**	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260C	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	TO-15	---	---	**X**	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	TO-15 SIM	---	---	**X**	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	8260C	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	TO-15	---	---	**X**	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	TO-15 SIM	---	---	**X**	ANAB
Dichlorophenol, 2,4-	120-83-2	8270C	**X**	**X**	---	ANAB
Dichlorophenol, 2,4-	120-83-2	8270D	**X**	**X**	---	ANAB
Dichloropropane, 1,2-	78-87-5	8260B	**X**	**X**	---	ANAB
Dichloropropane, 1,2-	78-87-5	8260C	**X**	**X**	---	ANAB
Dichloropropane, 1,2-	78-87-5	TO-15	---	---	**X**	ANAB
Dichloropropane, 1,2-	78-87-5	TO-15 SIM	---	---	**X**	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260B	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260C	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	TO-15	---	---	**X**	ANAB
Dieldrin	60-57-1	8081A	**X**	**X**	---	ANAB
Dieldrin	60-57-1	8081B	**X**	**X**	---	ANAB
Dieldrin	60-57-1	TO-10A	---	---	**X**	ORELAP
Diethyl Phthalate	84-66-2	8270C	**X**	**X**	---	ANAB
Diethyl Phthalate	84-66-2	8270D	**X**	**X**	---	ANAB
Dimethylphenol, 2,4-	105-67-9	8270C	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dimethylphenol, 2,4-	105-67-9	8270D	**X**	**X**	---	ANAB
Dimethylphthalate	131-11-3	8270C	**X**	**X**	---	ANAB
Dimethylphthalate	131-11-3	8270D	**X**	**X**	---	ANAB
Dinitrobenzene, 1,3-	99-65-0	8270C	**X**	**X**	---	ANAB
Dinitrobenzene, 1,3-	99-65-0	8270D	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270C	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270D	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8270C	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8270D	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8330A	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8330B	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270C	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270D	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8330A	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8330B	**X**	**X**	---	ANAB
Dinitrotoluene, 2-Amino-4,6-	35572-78-2	8330A	**X**	**X**	---	ANAB
Dinitrotoluene, 2-Amino-4,6-	35572-78-2	8330B	**X**	**X**	---	ANAB
Dinitrotoluene, 4-Amino-2,6-	19406-51-0	8330A	**X**	**X**	---	ANAB
Dinitrotoluene, 4-Amino-2,6-	19406-51-0	8330B	**X**	**X**	---	ANAB
Dioxane, 1,4-	123-91-1	8260B	**X**	**X**	---	ANAB
Dioxane, 1,4-	123-91-1	8260C	**X**	**X**	---	ANAB
Dioxane, 1,4-	123-91-1	TO-15	---	---	**X**	ANAB
Dioxane, 1,4-	123-91-1	TO-15 SIM	---	---	**X**	ANAB
Diphenylamine	122-39-4	8270C	**X**	**X**	---	ANAB
Diphenylamine	122-39-4	8270D	**X**	**X**	---	ANAB
Endosulfan (Endosulfan I + Endosulfan II)	115-29-7	8081A	**X**	**X**	---	ANAB
Endosulfan (Endosulfan I + Endosulfan II)	115-29-7	8081B	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Endosulfan I	959-98-8	8081A	**X**	**X**	---	ANAB
Endosulfan II	33213-65-9	8081A	**X**	**X**	---	ANAB
Endosulfan sulfate	1031-07-8	8081A	**X**	**X**	---	ANAB
Endrin	72-20-8	8081A	**X**	**X**	---	ANAB
Endrin	72-20-8	8081B	**X**	**X**	---	ANAB
Ethyl Chloride	75-00-3	8260B	**X**	**X**	---	ANAB
Ethyl Chloride	75-00-3	8260C	**X**	**X**	---	ANAB
Ethyl Chloride	75-00-3	TO-15	---	---	**X**	ANAB
Ethyl Chloride	75-00-3	TO-15 SIM	---	---	**X**	ANAB
Ethylbenzene	100-41-4	8260B	**X**	**X**	---	ANAB
Ethylbenzene	100-41-4	8260C	**X**	**X**	---	ANAB
Ethylbenzene	100-41-4	TO-15	---	---	**X**	ANAB
Ethylbenzene	100-41-4	TO-15 SIM	---	---	**X**	ANAB
Fluoranthene	206-44-0	8270C	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	8270C-SIM	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	8270D	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	8270D-SIM	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	TO-13A SIM	---	---	**X**	ANAB
Fluorene	86-73-7	8270C	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270C-SIM	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270D	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270D-SIM	**X**	**X**	---	ANAB
Fluorene	86-73-7	TO-13A SIM	---	---	**X**	ANAB
Heptachlor	76-44-8	8081A	**X**	**X**	---	ANAB
Heptachlor	76-44-8	8081B	**X**	**X**	---	ANAB
Heptachlor	76-44-8	TO-10A	---	---	**X**	ORELAP
Heptachlor Epoxide	1024-57-3	8081A	**X**	**X**	---	ANAB

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Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Heptachlor Epoxide	1024-57-3	8081B	**X**	**X**	---	ANAB
Heptachlor Epoxide	1024-57-3	TO-10A	---	---	**X**	ORELAP
Hexachlorobenzene	118-74-1	8270C	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270D	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270D-SIM	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260B	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260C	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8270C	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8270D	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	TO-15 SIM	---	---	**X**	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	TO-10A	---	---	**X**	ORELAP
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	TO-10A	---	---	**X**	ORELAP
Hexachlorocyclohexane, Delta- (δ-BHC)	319-86-8	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Delta- (δ-BHC)	319-86-8	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	TO-10A	---	---	**X**	ORELAP
Hexachlorocyclopentadiene	77-47-4	8270C	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270D	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270D-SIM	**X**	---	---	ANAB
Hexachloroethane	67-72-1	8270C	**X**	**X**	---	ANAB
Hexachloroethane	67-72-1	8270D	**X**	**X**	---	ANAB

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Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8330A	**X**	**X**	---	ANAB
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8330B	**X**	**X**	---	ANAB
Hexane, N-	110-54-3	8260B	**X**	**X**	---	ANAB
Hexane, N-	110-54-3	8260C	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260B	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260C	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270C	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270C-SIM	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270D	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270D-SIM	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	TO-13A SIM	---	---	**X**	ANAB
Isophorone	78-59-1	8270C	**X**	**X**	---	ANAB
Isophorone	78-59-1	8270D	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6010B	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6010C	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6020A	**X**	**X**	---	ANAB
Mercury (elemental)	7439-97-6	7470A	---	**X**	---	ANAB
Mercury (elemental)	7439-97-6	7471A	**X**	---	---	ANAB
Mercury (elemental)	7439-97-6	7471B	**X**	---	---	ANAB
Methoxychlor	72-43-5	8081A	**X**	**X**	---	ANAB
Methoxychlor	72-43-5	8081B	**X**	**X**	---	ANAB
Methoxychlor	72-43-5	TO-10A	---	---	**X**	ORELAP
Methyl Ethyl Ketone (2-Butanone)	78-93-3	8260B	**X**	**X**	---	ANAB
Methyl Ethyl Ketone (2-Butanone)	78-93-3	8260C	**X**	**X**	---	ANAB
Methyl Ethyl Ketone (2-Butanone)	78-93-3	TO-15	---	---	**X**	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	8260B	**X**	**X**	---	ANAB
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	8260C	**X**	**X**	---	ANAB
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	TO-15	---	---	**X**	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	8260B	**X**	**X**	---	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	8260C	**X**	**X**	---	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	TO-15	---	---	**X**	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	TO-15 SIM	---	---	**X**	ANAB
Methylene Chloride	75-09-2	8260B	**X**	**X**	---	ANAB
Methylene Chloride	75-09-2	8260C	**X**	**X**	---	ANAB
Methylene Chloride	75-09-2	TO-15	---	---	**X**	ANAB
Methylene Chloride	75-09-2	TO-15 SIM	---	---	**X**	ANAB
Methylnaphthalene, 1-	90-12-0	8270C	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270C-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270D	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270D-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	TO-13A SIM	---	---	**X**	ANAB
Methylnaphthalene, 2-	91-57-6	8270C	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270C-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270D	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270D-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	TO-13A SIM	---	---	**X**	ANAB
Naphthalene	91-20-3	8260B	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8260C	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270C	**X**	**X**	---	ANAB

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Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Naphthalene	91-20-3	8270C-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270D	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270D-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	TO-13A SIM	---	---	**X**	ANAB
Naphthalene	91-20-3	TO-15	---	---	**X**	ANAB
Naphthalene	91-20-3	TO-15 SIM	---	---	**X**	ANAB
Nickel, Total	7440-02-0	6010B	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	6010C	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	6020A	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8270C	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8270D	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8330A	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8330B	**X**	**X**	---	ANAB
Nitroglycerin	55-63-0	8330A	**X**	**X**	---	ANAB
Nitroglycerin	55-63-0	8330B	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270C	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270D	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270D-SIM	---	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270C	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270D	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270D-SIM	**X**	**X**	---	ANAB
Nitrosodiphenylamine, N-	86-30-6	8270C	**X**	**X**	---	ANAB
Nitrosodiphenylamine, N-	86-30-6	8270D	**X**	**X**	---	ANAB
Nitrotoluene, m-	99-08-1	8330A	**X**	**X**	---	ANAB
Nitrotoluene, m-	99-08-1	8330B	**X**	**X**	---	ANAB
Nitrotoluene, o-	88-72-2	8330A	**X**	**X**	---	ANAB
Nitrotoluene, o-	88-72-2	8330B	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Nitrotoluene, p-	99-99-0	8330A	**X**	**X**	---	ANAB
Nitrotoluene, p-	99-99-0	8330B	**X**	**X**	---	ANAB
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	8330A	**X**	**X**	---	ANAB
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	8330B	**X**	**X**	---	ANAB
Octyl Phthalate, di-N-	117-84-0	8270C	**X**	**X**	---	ANAB
Octyl Phthalate, di-N-	117-84-0	8270D	**X**	**X**	---	ANAB
PCB - Aroclor-1016	12674-11-2	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1221	11104-28-2	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1232	11141-16-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1242	53469-21-9	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1248	12672-29-6	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1254	11097-69-1	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1260	11096-82-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1262	37324-23-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1268	11100-14-4	8082A	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270C	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270C-SIM	**X**	---	---	ANAB
Pentachlorophenol	87-86-5	8270D	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270D-SIM	**X**	---	---	ANAB
Pentaerythritol tetranitrate (PETN)	78-11-5	8330A	**X**	**X**	---	ANAB
Pentaerythritol tetranitrate (PETN)	78-11-5	8330B	**X**	**X**	---	ANAB
Perfluorooctane Sulphonic Acid (PFOS)	1763-23-1	537	**X**	**X**	---	ANAB
Perfluorooctanoic acid (PFOA)	335-67-1	537	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270C	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270C-SIM	**X**	**X**	---	ANAB

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Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Phenanthrene	85-01-8	8270D	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270D-SIM	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	TO-13A SIM	---	---	**X**	ANAB
Phenol	108-95-2	8270C	**X**	**X**	---	ANAB
Phenol	108-95-2	8270D	**X**	**X**	---	ANAB
Propyl benzene	103-65-1	8260B	**X**	**X**	---	ANAB
Propyl benzene	103-65-1	8260C	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270C	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270C-SIM	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270D	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270D-SIM	**X**	**X**	---	ANAB
Pyrene	129-00-0	TO-13A SIM	---	---	**X**	ANAB
Selenium	7782-49-2	6010B	**X**	**X**	---	ANAB
Selenium	7782-49-2	6010C	**X**	**X**	---	ANAB
Selenium	7782-49-2	6020A	**X**	**X**	---	ANAB
Silver	7440-22-4	6010B	**X**	**X**	---	ANAB
Silver	7440-22-4	6010C	**X**	**X**	---	ANAB
Silver	7440-22-4	6020A	**X**	**X**	---	ANAB
Styrene	100-42-5	8260B	**X**	**X**	---	ANAB
Styrene	100-42-5	8260C	**X**	**X**	---	ANAB
Styrene	100-42-5	TO-15 SIM	---	---	**X**	ANAB
TCDD, 2,3,7,8-	1746-01-6	8290A	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260B	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260C	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260B	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260C	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260C	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Tetrachloroethane, 1,1,2,2-	79-34-5	TO-15	---	---	**X**	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	TO-15 SIM	---	---	**X**	ANAB
Tetrachloroethylene	127-18-4	8260B	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	8260C	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	TO-15	---	---	**X**	ANAB
Tetrachloroethylene	127-18-4	TO-15 SIM	---	---	**X**	ANAB
Tetryl (Trinitrophenylmethylnitramine)	479-45-8	8330A	**X**	**X**	---	ANAB
Tetryl (Trinitrophenylmethylnitramine)	479-45-8	8330B	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6010B	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6010C	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6020A	**X**	**X**	---	ANAB
Toluene	108-88-3	8260B	**X**	**X**	---	ANAB
Toluene	108-88-3	8260C	**X**	**X**	---	ANAB
Toluene	108-88-3	TO-15	---	---	**X**	ANAB
Toluene	108-88-3	TO-15 SIM	---	---	**X**	ANAB
Toxaphene	8001-35-2	8081A	**X**	**X**	---	ANAB
Toxaphene	8001-35-2	8081B	**X**	**X**	---	ANAB
Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	76-13-1	8260B	**X**	**X**	---	ANAB
Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	76-13-1	8260C	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,3-	87-61-6	8260B	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,3-	87-61-6	8260C	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	8260B	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	8260C	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	TO-15	---	---	**X**	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	TO-15 SIM	---	---	**X**	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Trichloroethane, 1,1,1-	71-55-6	8260B	**X**	**X**	---	ANAB
Trichloroethane, 1,1,1-	71-55-6	8260C	**X**	**X**	---	ANAB
Trichloroethane, 1,1,1-	71-55-6	TO-15	---	---	**X**	ANAB
Trichloroethane, 1,1,1-	71-55-6	TO-15 SIM	---	---	**X**	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260B	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260C	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	TO-15	---	---	**X**	ANAB
Trichloroethane, 1,1,2-	79-00-5	TO-15 SIM	---	---	**X**	ANAB
Trichloroethylene	79-01-6	8260B	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	8260C	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	TO-15	---	---	**X**	ANAB
Trichloroethylene	79-01-6	TO-15 SIM	---	---	**X**	ANAB
Trichlorofluoromethane	75-69-4	8260B	**X**	**X**	---	ANAB
Trichlorofluoromethane	75-69-4	8260C	**X**	**X**	---	ANAB
Trichlorofluoromethane	75-69-4	TO-15	---	---	**X**	ANAB
Trichlorofluoromethane	75-69-4	TO-15 SIM	---	---	**X**	ANAB
Trichlorophenol, 2,4,5-	95-95-4	8270C	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,5-	95-95-4	8270D	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270C	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270D	**X**	**X**	---	ANAB
Trichloropropane, 1,2,3-	96-18-4	8260B	**X**	**X**	---	ANAB
Trichloropropane, 1,2,3-	96-18-4	8260C	**X**	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	8260B	**X**	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	8260C	**X**	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	TO-15	---	---	**X**	ANAB
Trimethylbenzene, 1,3,5-	108-67-8	8260B	**X**	**X**	---	ANAB
Trimethylbenzene, 1,3,5-	108-67-8	8260C	**X**	**X**	---	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Trimethylbenzene, 1,3,5-	108-67-8	TO-15	---	---	**X**	ANAB
Trinitrobenzene, 1,3,5-	99-35-4	8270C	**X**	**X**	---	ANAB
Trinitrobenzene, 1,3,5-	99-35-4	8270D	**X**	**X**	---	ANAB
Trinitrobenzene, 1,3,5-	99-35-4	8330A	**X**	**X**	---	ANAB
Trinitrobenzene, 1,3,5-	99-35-4	8330B	**X**	**X**	---	ANAB
Trinitrotoluene, 2,4,6-	118-96-7	8330A	**X**	**X**	---	ANAB
Trinitrotoluene, 2,4,6-	118-96-7	8330B	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6010B	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6010C	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6020A	**X**	**X**	---	ANAB
Vinyl Acetate	108-05-4	8260B	**X**	**X**	---	ANAB
Vinyl Acetate	108-05-4	8260C	**X**	**X**	---	ANAB
Vinyl Acetate	108-05-4	TO-15	---	---	**X**	ANAB
Vinyl Chloride	75-01-4	8260B	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	8260C	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	TO-15	---	---	**X**	ANAB
Vinyl Chloride	75-01-4	TO-15 SIM	---	---	**X**	ANAB
Xylene, m-	108-38-3	TO-15	---	---	**X**	ANAB
Xylene, m-	108-38-3	TO-15 SIM	---	---	**X**	ANAB
Xylene, m+p -	-	8260B	**X**	**X**	---	ANAB
Xylene, m+p -	-	8260C	**X**	**X**	---	ANAB
Xylene, o-	95-47-6	8260B	**X**	**X**	---	ANAB
Xylene, o-	95-47-6	8260C	**X**	**X**	---	ANAB
Xylene, o-	95-47-6	TO-15	---	---	**X**	ANAB
Xylene, o-	95-47-6	TO-15 SIM	---	---	**X**	ANAB
Xylene, p-	106-42-3	TO-15	---	---	**X**	ANAB
Xylene, p-	106-42-3	TO-15 SIM	---	---	**X**	ANAB

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Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Xylene, Total	1330-20-7	8260C	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6010B	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6010C	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6020A	**X**	**X**	---	ANAB
Gasoline Range Organics (C6 – C10)	N/A	AK 101	**X**	**X**	---	ANAB
Diesel Range Organics (C10 – C25)	N/A	AK 102	**X**	**X**	---	ANAB
Residual Range Organics (C25 – C36)	N/A	AK 103	**X**	**X**	---	ANAB
TCLP Extraction	N/A	1311	**X**	**X**	---	ANAB
SPLP	N/A	1312	**X**	**X**	---	ANAB
Acid Digestion for Metals Analysis	N/A	3010A	---	**X**	---	ANAB
Acid Digestion	N/A	3050B	**X**	**X**	---	ORELAP
Microwave Assisted Acid Digestion	N/A	3050B	**X**	---	---	ANAB
Separatory Funnel Extraction	N/A	3510C	---	**X**	---	ANAB
Soxhlet Extraction	N/A	3540C	**X**	---	---	ANAB
Ultrasonic Extraction	N/A	3550B	**X**	---	---	ANAB
Ultrasonic Extraction	N/A	3550C	**X**	---	---	ANAB
Florisil Cleanup	N/A	3620B	**X**	**X**	---	ORELAP
Florisil Cleanup	N/A	3620C	**X**	**X**	---	ANAB
Sulfur cleanup	N/A	3660B	---	**X**	---	ANAB
Purge and Trap	N/A	5030B	**X**	**X**	---	ANAB
Purge and Trap	N/A	5030C	---	**X**	---	ANAB
Closed System Purge and Trap	N/A	5035	**X**	**X**	---	ANAB
Closed System Purge and Trap	N/A	5035A	**X**	**X**	---	ANAB
Mercury Digestion	N/A	7470A	---	**X**	---	ORELAP
Mercury Digestion	N/A	7471A	**X**	---	---	ANAB

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Title: Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified), Method PFAS by LCMSMS Compliant with QSM 5.1 Table B-15]

Approvals (Signature/Date):

02/22/2019	02/25/2019
Robert Hrabak Date	Joe Schairer Date
Technical Manager	Health & Safety Manager / Coordinator
02/25/2019	02/26/2019
Lisa Stafford Date	Chris Williams Date
Quality Assurance Manager	Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment, and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

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	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 (non-routine analyte)	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid (non-routine analyte)	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-1
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-nonanesulfonic acid	PFNS	8789-57-2
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluoro-1-dodecansulfonic 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-perfluorohexane sulfonate *(4:2)*	4:2 FTS	757124-72-4

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Compound Name	Abbreviation	CAS #
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4
1H,1H,2H,2H-perfluorododecane sulfonate *(10:2)*	10:2 FTS	120226-60-0

Abbreviations in parenthesis are the abbreviations listed in Method 537, where they differ from the abbreviation used by the laboratory's LIMS.

1.2. Additional analytes supported by this method: The following analytes can be supported by this method under special request.

Compound Name	Abbreviation	CAS #
Fluorinated Replacement Chemicals		
Dona (Donic acid)	Dona	919005-14-4
Perfluoro(2-propoxypropanoic) acid	HFPO-DA or GenX	13252-13-6
F53B (reported as the summation of the following)	F53B	NA
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonate	F53B major	73606-19-6
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonate	F5B minor	83329-89-9

1.3. 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 ug/kg – 2.0 ug/kg	0.2 ug/kg - 40 ug/kg
Tissue	1 g	1.0 ug/kg – 10 ug/kg	1.0 ug/kg – 200 ug/kg

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1.4. The procedure for the analysis of water samples via in line solid phase extraction (SPE) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a [redacted] is described in Attachment 1 of this SOP.

1.5. This procedure also includes direction for preparing and analyzing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk.

1.6. When undertaking projects for the Department of Defense (DoD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an [redacted] solution.

2.2. Soil/sediment/tissue samples are extracted with a [redacted] solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using [redacted]. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

2.4. An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compounds 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 quantitated by the IDA method using a closely related labeled analog.

2.5. Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

2.6. Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to oxidation with [redacted] prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated.

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3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonic acids

3.3. FOSA: Perfluorinated sulfonamide

3.4. PFOA: Perfluorooctanoic acid

3.5. PFOS: Perfluorooctane sulfonic acid

3.6. PTFE: Polytetrafluoroethylene (e.g., Teflon®)

3.7. SPE: Solid phase extraction

3.8. PP: Polypropylene

3.9. PE: Polyethylene

3.10. HDPE: High density polyethylene

3.11. AFFF: Aqueous Film Forming Foam

3.12. IDA: Isotope dilution analyte

3.13. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. 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.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. 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.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. Random evaporation losses have been observed with the polypropylene 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.

4.3.3. 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 polypropylene screw caps.

4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with

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similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PFBS, EtFOSAA, and MeFOSAA based upon the scientific literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolve or not, but usually with a deflection point resolved during peak integration. The later of these peaks matches the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

As of this writing, only PFOS, PFOA, and PFHxS 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.

4.6. In an attempt to reduce PFOS bias, it is required that *m/z* 499>80 transition be used as the quantitation transition.

4.7. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.30 ng/L or 0.015 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

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5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS and PFAS samples must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. 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.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware 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.

5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), 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.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. 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.

5.1.7. Glass containers are not to be used for “tumbling” soil samples.

5.2. Primary Materials Used

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

Material[1]	**Hazards**	**Exposure Limit**[2]	**Signs and Symptoms of Exposure**
Acetic Acid (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.

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Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
[redacted] (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.
[redacted] (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.
Sodium Hydroxide (3-0-1)	Corrosive Poison	2 mg/cm^3 (Ceiling)	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.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

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6. EQUIPMENT AND SUPPLIES

6.1. 15 mL polypropylene test tubes with polypropylene screw caps.

6.2. 50 mL graduated plastic centrifuge tubes.

6.3. 125 mL HDPE bottles with HDPE screw caps.

6.4. 250 mL HDPE bottles with HDPE screw caps. The average weight of the HDPE bottles with HDPE screw caps are calibrated once per year. The calibration is performed by weighing 10 bottles with caps and dividing by 10 to get the average weight. The average weight is used in section (11.3.5.1.d).

6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.6. Extract concentrator or nitrogen manifold with water bath heating to 50-55°C.

6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300 µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.

6.9. SPE columns

6.9.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH, Waters SepPak C18, 1 to 10g, or equivalent.

6.9.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.

6.9.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFAS from aqueous sample.

[redacted]

6.10. Graphitized carbon (Envi-Carb™ or equivalent).

6.11. Vacuum manifold for Solid Phase Extraction (SPE).

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6.12. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.13. Water bath: Heated with concentric ring cover capable of temperature control (±5°C) up to 95°C. The bath must be used in a fume hood.

6.14. Plastic tub for an ice bath, AKRO-N.S.T. part No. 35-180 or equivalent.

6.15. pH indicator paper, wide range.

6.16. Bottle rotating apparatus for soil extractions.

6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.

6.18. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. Both HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. 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.

6.18.1. [redacted] LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a [redacted] Triple Quad MS. The instrument control and data acquisition software is [redacted] or equivalent.

6.18.1.1. [redacted] HPLC equipped with [redacted] pumps and [redacted] degassing unit or equivalent.

6.18.1.2. [redacted], or equivalent.

6.18.1.3. PFAS Isolator column, [redacted]. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.18.2. [redacted] LC/MS/MS

This consists of a Waters Acquity UPLC system interfaced with a [redacted] tandem mass spectrometer. The instrument control and data acquisition software is [redacted], or equivalent.

6.18.2.1. Analytical column: [redacted]

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6.18.2.2. PFAS Isolator column, [redacted] or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.19. 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. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes. Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use.	**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 corona and multiplier are functioning.
Semi-Annually Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable	**Annually** Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

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7. REAGENTS AND STANDARDS

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

7.1.1. Acetic acid, glacial

7.1.2. [redacted] The resultant solution is filtered through a 0.22um filter before use. This solution has volatile components, thus it should be replaced every 7 days or sooner.

7.1.3. [redacted].

7.1.4. Hexane

7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water

7.1.6. Hydrochloric acid (HCl), concentrated, reagent grade

7.1.7. Methanol

7.1.8. [redacted].

7.1.9. [redacted], reagent grade

7.1.10. Ottawa Sand

7.1.11. Sodium hydroxide (NaOH), 0.1N, in water: Prepared by diluting 400mL of 1N NaOH into 3.6L of water for a total volume of 4L.

7.1.12. Sodium hydroxide (NaOH), 10N, reagent grade

7.1.13. Water, Nanopure or Millipore, must be free of interference and target analytes.

7.2. Standards

7.2.1. PFAS are purchased as high purity solids (96% or greater) or as certified solutions. 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.

7.2.1.1. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.30 ng/L or 0.015 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

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7.2.2. 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.

7.2.3. 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 $Mass_{salt}$ × MW_{acid} / MW_{salt}

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

7.2.4. 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 adjusted by a factor of 0.956.

7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of stock solutions in 80% methanol/water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluoroalkylcarboxylic acids (PFCAs)							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonic acids (PFSAs)							

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Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
PFBS	0.5	1.0	5.0	20	50	200	400
PFPeS	0.5	1.0	5.0	20	50	200	400
PFHxS *	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS *	0.5	1.0	5.0	20	50	200	400
PFNS	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400
PFDoS	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamides (FOSA)							
FOSA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
Fluorotelomer sulfonates (FTS)							
4:2 FTS	0.5	1.0	2.0	20	50	200	400
6:2 FTS	0.5	1.0	5.0	20	50	200	400
8:2 FTS	0.5	1.0	5.0	20	50	200	400
10:2 FTS	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes (IDA)							
13C4-PFBA	50	50	50	50	50	50	50
13C5-PFPeA	50	50	50	50	50	50	50
13C2-PFHxA	50	50	50	50	50	50	50
13C4-PFHpA	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50
13C2-PFDA	50	50	50	50	50	50	50
13C2-PFUdA	50	50	50	50	50	50	50
13C2-PFDoA	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50
13C3-PFBS	50	50	50	50	50	50	50
13C2-PFTeDA	50	50	50	50	50	50	50
13C2-PFHxDA	50	50	50	50	50	50	50
13C8-FOSA	50	50	50	50	50	50	50
d5-EtFOSAA	50	50	50	50	50	50	50
d3-MeFOSAA	50	50	50	50	50	50	50
M2-4:2FTS ǂ	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50
Internal Standard (IS)							

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Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
13C2-PFOA	50	50	50	50	50	50	50

** Both branched and linear isomers are used.*

ǂ - This compound is used as a reverse surrogate for the TOP analysis.

***Note**: Sample extracts are in 80% MeOH/H $_2$O.*

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Fluorinated Replacement Chemicals							
HFPO-DA	0.5	1.0	5.0	20	50	200	400
9CI-PF3ONS (F53B major)	0.5	1.0	5.0	20	50	200	400
11CI-PF3OUdS (F53B minor)	0.5	1.0	5.0	20	50	200	400
Dona	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes							
13C3-HFPO-DA	0.5	1.0	5.0	20	50	200	400

***Note**: Sample extracts are in 80% MeOH/H $_2$O.*

***Note**: The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program. The concentration of the calibration solutions for non-concentrated extracts is 1/20th the levels indicated above.*

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7.4.1. A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard This technical (qualitative) grade PFOA standard is analyzed initially, after every initial calibration or when significant changes are made to the HPLC parameters.

7.4.1.1. Attach this document to the ICV from the associated ICAL by scanning the document and associating it to the file as a document type of High Res MS Tune in TALS. Use the following naming convention: "_ZbatchnumberTPFOA".

7.5. Initial Calibration Verification Standard (ICV)

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. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, 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 IS are added at a fixed concentration of 50 ng/mL.

7.6. LCS/Matrix PFC Spike Solution, 20 ng/mL

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 20 ng/mL in methanol.

7.7. PFC Isotope Dilution Analyte Solution, 50 ng/mL

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each compound at a concentration of 50 ng/mL in methanol.

7.8. Reverse Surrogate Solution, 1000 ng/mL

The reverse surrogate solution is prepared by diluting M2-4:2 FTS to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all samples for the TOP assay to monitor the efficiency of the oxidation process.

7.9. Internal Standard Solution, 250 ng/mL

The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 250 ng/mL in methanol. This is added to all extracts prior to analysis. The internal standard solution used for the non-concentrated extracts is at a concentration of 50 ng/mL.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Soil samples are collected in pre-cleaned 8 oz. HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.1.1. Water samples collected from a known chlorinated source should be preserved with Trizma.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples must be extracted within 14 days of collection. Soil samples must also be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at 0 - 6°C, and analyzed within 40 days from extraction.

8.2.1. Projects performed for the state of New Jersey have an analytical holding time 28 days from the extraction date.

8.2.2. For projects performed for the state of New Jersey a field reagent blank (FRB) must be collected with each sample set. Acceptance limits are <RL for each analyte.

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***Note**: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted time stability studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14/40 day holding times given above are based on the stability study and general EPA convention for the holding time of extractable organic compounds in water and soil.*

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. 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. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid samples, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and then implemented when target analytes are detected in the method blank above the reporting limit or when IDA recoveries are outside of the control limits. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

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9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit/LOQ for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Reprepare and reanalyze all field and QC samples associated with the contaminated method blank.

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9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC, and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

9.4.1. Projects performed for the state of New Jersey: LCS (mid and high spike) recovery limits are 70-130%. Low level LCS recovery limits are 50-150%. The spike level must rotate between low, medium and high.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An

MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, and then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.

9.5.1. Projects performed for the state of New Jersey: MS/MSD (mid and high spike) recovery limits are 70-130%. Low level MS/MSD recovery limits are 50-150%. The spike level must rotate between low, medium and high.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.7. Initial calibration verification (ICV) –A second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.
- Rerun the initial calibration.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). 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-nose ratio is greater than 10:1.

9.8.2.1. 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.

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9.8.2.2. 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%.

9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.8.2.3. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for IDA recoveries which are 50-150%. If QC or field samples do not meet these criteria then re-extraction is required.

9.9. Internal Standard

9.9.1. The Internal Standard (IS) is added to each field and QC samples prior to analysis. The CCV IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

9.9.2. Sample IS response (peak area) must be within ±50% of the response (peak area) in the most recent CCV.

9.9.3. If the IS does not meet criteria, re-analyze the extract. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration.

9.10. TOP Oxidation Efficiency

9.10.1. If the data indicates incomplete oxidation (i.e. the Post-TOP M2-4:2 FTS recovery is greater than 10% or the Post-TOP precursor concentration is greater than 10% of the Pre-TOP concentration) then a second aliquot (10 mL or a 0.2g equivalent) should be processed.

9.10.2. A reduced sample size may be used initially if sample history or other information indicates the sample is grossly contaminated.

9.11. Ion Ratio

9.11.1. Compare the quantifier/qualifier SRM transition ratio in the sample to the SRM transition ratio in the standard.

9.11.2. The quantifier/qualifier SRM ion ratio should be within + 50% of the average of the quantifier/qualifier SRM ion ratios calculated from the midlevel ICAL point or from the CCV, if an ICAL is not run.

9.11.3. At this time the ion ratio evaluation is a quantitative identification tool.

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Analyst judgement should be used if the ratio does not meet criteria. Data should be qualified "I" if the ratio is not met.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 "Calibration Curves and Selection of Calibration Points".

10.2. Routine instrument operating conditions are listed in the table in Section 11.18.

10.3. 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 mobile phase using a tee fitting at a point just before the entrance to the 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 in Section 11.18.

10.3.1. Once the optimal mass assignments (within ±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion. For QSM work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to, new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. 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.

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10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Initial Calibration

10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.8.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.

10.8.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.

10.8.2. 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.

10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against an identically labeled analog must be < 35% for the curve to be valid.

10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against a closely related labeled analog IDA must be < 50% for the curve to be valid.

10.8.2.3. 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).

10.8.2.4. The Internal Standard (IS) response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

10.8.2.5. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for initial calibration: The %RSD of the RFS for all analytes must be <20%. Linear or non-linear calibrations must have r^2>0.99 for each analyte. Analytes must be within 70-130% of their true value for each calibration standard.

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10.8.2.6. Projects performed for the state of New Jersey: Each calibration point, except the lowest point, of each analyte should be calculated to be within 70-130% of the true value. The lowest calibration point that is at or below the MRL should be within 50-150% of its true value.

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.9.2. 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

10.9.3. 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.

10.9.4. 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.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.5. 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

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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.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank containing both IDA and IS.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. 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.

10.10.4. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for instrument blanks. One is required immediately following the highest standard analyzed and *daily prior to sample analysis*. The instrument blank must be < ½ the LOQ.

10.11. Initial Calibration Verification (ICV)

10.11.1. 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.

10.11.2. The recovery for the ICV must meet the appropriate following criteria:

10.11.2.1. The native analyte must be within or equal to 60-140% for all native analytes quantitated against an identically labeled analog IDA.

10.11.2.2. The native analyte must be within or equal to 50-150% for all native analytes quantitated against a closely related labeled analog IDA.

10.11.2.3. The IDA must be within or equal to 50-150%.

10.11.3. Projects performed under the auspices of the DoD/DOE QSM (Version 5.1) and the state of New Jersey must meet these criteria for the ICV: Analyte concentrations must be within ±30% of their true values for all analytes, IDA and target.

10.11.4. See Section 9.7 for corrective actions in the event that the ICV does not meet

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the criteria above.

10.12. 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 from low level (LOQ/RL) to mid level. 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.

10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated against an identically labeled analog and equal to or within 50% to 150% for all natives quantitated against a closely related labeled analog. The recovery for the IDA must be within or equal to 50-150%.

10.12.2. The Internal Standard (IS) response (peak area) must be within ± 50% from the response (peak area) from the midpoint of the initial calibration.

10.12.2.1. Sample IS response (peak area) must be within ± 50% of the response (peak area) in the most recent CCV.

10.12.3. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

10.12.4. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for CCV. All analyte concentrations must be within ± 30% of their true value. Additionally, prior to analysis and at least once every 12 hours an instrument sensitivity check (ISC/CCVL) must be analyzed. The analyte concentrations must be at LOQ and the concentrations must be within ± 30% of their true value. This can be used as a CCV.

10.12.5. Projects performed for the state of New Jersey: All analyte concentrations in the CCV must be within + 30% of their true value. All analyte concentrations in the low level CCV must be within + 50% of their true value.

11. PROCEDURE

11.1. 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 an Non-Conformance Memo (NCM). The NCM process

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is described in more detail in SOP WS-QA-0023. 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.

11.2. Water Sample Preparation

11.2.1. Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action. Decanting or filtering of the sample can lead to a low bias.

11.2.2. If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank 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.

11.2.3. Weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume. Unless otherwise directed by client, use the entire sample volume.

11.2.4. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.2.5. Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS.

11.2.6. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L.

11.2.7. Add 0.5 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.3. Solid Phase Extraction (SPE) of Aqueous Samples

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.

11.3.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.

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Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

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.

11.3.2. Wash with 5.0 mL of [redacted].

11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.

11.3.4. Appropriately label the columns and add the reservoir to the column.

11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at a rate of approximately 2 to 5 drops per second.

11.3.5.1. If the SPE column should plug (flow rate <1 drop per minute) prior to the entire content of the sample container passing through the column do the following:

1. Stop adding sample to the reservoir.
2. Return any remaining sample volume back to the original container.
3. Weigh the original container and record this weight into the worksheet notes field within the TALS extraction batch.
4. Determine the full volume of sample fortified by using the "Gross Weight" – (remaining sample volume – default tare weight of a sample container (26.1 g)).
5. Enter this value into the "Initial Amount" field in the TALS extraction batch.
6. Proceed to Section 11.4, noting that additional vacuum or pressure might be needed to elute the SPE column.

11.3.6. After the entire sample has been loaded onto the column, rinse the sample bottle with two 5 mL aliquots of reagent water and pour onto the column reservoir.

11.3.7. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.3.8. After the sample and water rinse have completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

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11.4. SPE Column Wash of Aqueous Samples with Hexane

11.4.1. Load the first 5 mL of hexane to soak for five minutes and then elute to waste.

11.4.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.4.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

11.5.1. Rinse sample bottles with 5 mL of [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of [redacted]. The total collection should be approximately 10 mL.

11.5.3. **Note: If the extracts will not be concentrated elute extract with a total of 8 mL of [redacted].**

11.5.4. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This required for all DoD/DOE extracts.

11.6. Extract Concentration for Aqueous Extracts (Note, if the extract will not be concentrated, proceed to Section 11.7.)

11.6.1. Prior to concentrating each sample, add 100 uL of water.

11.6.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1. This blow down must take a minimum of 3.5 hours.

11.6.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.6.3. Add 300 uL of methanol and mix the contents well using a vortex mixer.

11.6.4. Add 100 uL of Internal Standard (IS) 250 ng/mL concentration solution to each extract and vortex to mix.

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11.6.5. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.6.6. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.6.7. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.7. Final volume for non-concentrated extract

11.7.1. If the extract does not undergo concentration add 0.5 mL of IS 50 ng/mL concentration and 2 mL of water to the extract. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.7.1.1. Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step.

11.7.2. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.7.3. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps cannot be used due to detection of low level concentration of PFAS.

11.8. Soil, Sediment and Tissue Sample Preparation and Extraction

11.8.1. Visually inspect soil samples for homogeneity.

11.8.1.1. Projects performed under the auspices of the DoD/DOE must have the entire sample homogenized prior to subsampling in accordance with QSM 5.1 criteria (see SOP WS-QA-0018).

11.8.2. Weigh a representative 5 g aliquot of soil, sediment or 1 g of tissue sample into a 50 mL HDPE wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested.

11.8.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand or 0.1 g of oil.

11.8.4. Spike the LCS and MS/MSD (if requested) with 1.0 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.

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11.8.4.1. Spike non-concentrated samples at 0.5 mL of LCS/Matrix PFC Spike Solution.

11.8.5. Add 1.0 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.8.5.1. Spike non-concentrated samples at 0.5 mL of IDA PFC Solution.

11.8.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

11.8.7. Add 20 mL of [redacted] to each sample.

11.8.8. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.8.9. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.8.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.8.11. Collect and decant the [redacted] extract to a new 50 mL centrifuge tube.

11.8.12. Add another 2 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.8.13. Combine the rinsate to the first corresponding tubes.

11.8.14. To the final [redacted] extract, add 2 mL of water to each.

11.8.15. Concentrate the [redacted] extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume.

11.8.16. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.

11.8.17. Centrifuge at 3500 rpm for 15 minutes.

11.9. Solid Extract Cleanup by SPE

Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.

11.9.1. Condition the SPE cartridges by passing the following without drying the column.

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Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

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.

11.9.2. Wash with 5.0 mL of [redacted]

11.9.3. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.9.4. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.9.5. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.9.6. Dry the columns with vacuum for 15 minutes.

11.10. SPE Column Wash of Solid Extracts with Hexane

11.10.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

11.10.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.10.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.11. SPE Elution of Solid Extracts – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.11.1. Rinse extraction bottles with 5 mL of [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.11.2. Repeat extract bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of [redacted]. The total collection should be approximately 10 mL.

11.11.3. **Note: If the extracts will not be concentrated elute extract with a total of 8 mL of [redacted].**

11.11.4. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This is

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required for all DoD/DOE extracts.

11.12. Extract Concentration for Solid Samples (Note, if the extract will not be concentrated, proceed to Section 11.7)

11.12.1. Prior to concentrating each sample, add 200 uL of water.

11.12.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains.

11.12.2.1. This blow down must take a minimum of 3.5 hours.

11.12.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.12.2.3. Add 600 uL of methanol and mix the contents well using a vortex mixer.

11.12.2.4. Add 200 uL of Internal Standard (IS) 250 ng/mL concentration solution to each extract and vortex to mix.

11.12.3. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.12.4. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.*

11.13. Product/Dispersion Samples

11.13.1. Check the solubility of the material in both methanol and water

11.13.1.1. If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water extraction procedures). Fortify sample appropriately with IDA or PFC spike solution, see Section 11.2.

11.13.1.2. If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (MeOH).

11.13.1.2.1. If the material does not completely dissolve, contact your immediate supervisor.

11.13.2. Take 100 uL of the 10 mL solution and dilute it to 10 mL in MeOH.

11.13.3. Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 0.5 mL of labeled IDA

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solution (Section 7.7).

11.13.4. DO NOT PASS EXTRACT THROUGH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.13.5. Proceed to Section 11.6 of this SOP for extract concentration.

11.14. TOP (Total Oxidizable Precursor) Assay for Aqueous Samples

11.14.1. Prepare 3-250 mL HDPE containers with HPLC grade water to create the needed QC Samples (MB, LCS/LCSD).

11.14.2. Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.14.3. Spike the “Pre” and “Post” MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.14.4. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution (Section 7.6), both regular and “add-on”, and 25 uL of the reverse surrogate solution (Section 7.8).

11.14.5. Remove the methanol solvent from all Post QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.14.6. Add 2g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.

11.14.7. Subsample 100 mL aliquots of water from each field sample and QC from the 250 mL containers into each of the corresponding 125 mL containers for both the “Pre” and “Post” samples. Spike all “Pre” and “Post” samples with 25uL of the reverse surrogate solution (Section 7.8).

11.14.8. Set aside all “Pre” sample containers.

11.14.9. Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.

11.14.10. Add 2 g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.

11.14.11. Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.14.12. After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

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11.14.13. Adjust the pH of "Post" samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.14.14. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 7.7), both regular and add-on.

11.14.15. Use the following SPE procedure for both "Pre" and "Post" samples:

11.14.15.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.14.15.2. Establish a sample loading flow rate of 3-5 drops per second for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.14.15.3. Wash/condition the SPE column with 5 mL of [redacted], then 5 mL water.

11.14.15.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 3-5 drops per second.

11.14.15.5. Add 5 mL rinse water

11.14.15.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.14.15.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.14.15.8. Allow the column to dry well using vacuum for 5 minutes. Columns must be dry before continuing.

11.14.15.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of [redacted], and add to the SPE cartridge as eluent.

11.14.15.10. Repeat with another 5 mL of [redacted].

11.14.15.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

11.15. TOP (Total Oxidizable Precursor) Assay for Soil Samples

11.15.1. Weigh representative 2 g aliquots of soil for each "Pre" and "Post" sample into a 50 mL centrifuge tube.

11.15.2. For the method blank and LCS matrix, use 2 g each of Ottawa sand for each

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"Pre" and "Post" QC sample.

11.15.3. Add 20 mL of [redacted] to each sample.

11.15.4. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.15.5. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.15.6. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.15.7. Collect and decant the [redacted] extract to a new 50 mL centrifuge tube.

11.15.8. Add another 2 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.15.9. Combine the rinsate to the first corresponding tubes.

11.15.10. Proceed to Section 11.16.2 (Envi-carb clean up)

11.15.11. To the final [redacted] extract, add 0.5 mL of water to each.

11.15.12. Concentrate the [redacted] extract under nitrogen to less than 0.25 mL.

11.15.13. Dilute extract up to 50 mL with water in the centrifuge tube and vortex.

11.15.14. Prepare enough 125 mL HDPE containers as needed for all "Pre" and "Post" samples, including QC. Label each appropriately.

11.15.15. Spike the "Pre" and "Post" MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.15.16. Spike the "Pre" and "Post" LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution and 25 uL of the reverse surrogate solution (Section 7.8).

11.15.17. Remove the methanol solvent from all "Post" QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.15.18. Add 2g of [redacted] and 1.9 mL of [redacted] to each "Post" sample container.

11.15.19. Transfer extract from the centrifuge tube to the appropriate 125 mL

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container.

11.15.20. Rinse the centrifuge container with an additional 50 mL of water and transfer to the appropriate 125 mL container.

11.15.21. Set aside all “Pre” sample containers.

11.15.22. Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.

11.15.23. Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.15.24. After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

11.15.25. Adjust the pH of “Post” samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.15.26. Spike both “Pre” and “Post” samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 7.7).

11.15.27. Use the following SPE procedure for both “Pre” and “Post” samples:

11.15.27.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.15.27.2. Establish a sample loading flow rate of 3-5 drops per second for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.15.27.3. Wash/condition the SPE column with 5 mL of [redacted], then 5 mL water.

11.15.27.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 3-5 drops per second.

11.15.27.5. Add 5 mL rinse water

11.15.27.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.15.27.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.15.27.8. Allow the column to dry well using vacuum for 5 minutes. Columns must be dry before continuing.

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11.15.27.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of [redacted], and add to the SPE cartridge as eluent.

11.15.27.10. Repeat with another 5 mL of [redacted].

11.15.27.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

Note: *If the extracts will not be concentrated elute extract with a total of 8 mL (2 4 mL rinses) of* [redacted].

11.16. Other Types of Sample Cleanup

11.16.1. Freezing technique to remove lipids.

If samples contain lipids then freeze the methanolic extract and QC extracts at -20°C for at least 1 hour. Collect the solvent layer.

11.16.2. Cleanup with graphitized carbon will be applied to all samples as needed but is required for all DoD/DOE extracts.

11.16.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.16.2.2. Shake vigorously and then let sit for 10 minutes.

11.16.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.16.2.4. Decant the solvent layer.

11.16.2.5. Proceed to Section 11.6, 11.7 or 11.12 as applicable.

11.17. AFFF Sample Preparation

11.17.1. QC for AFFF samples consists of a method blank, a laboratory control sample and a sample or matrix duplicate only. No matrix spike or matrix spike duplicate is needed.

11.17.2. Perform a 1,000,000 X serial dilution of the AFFF sample. Dilute 1 mL of AFFF sample to 1L with laboratory supplied water. Then dilute 1mL of this dilution to 1L with laboratory supplied water.

11.17.2.1. Be sure to retain all dilutions should the initial analysis warrant re-analysis at higher concentration.

11.17.3. Subsample 2.0 mL of this dilution and fortify with 0.5 mL IDA solution and 0.5mL of IS (50 ng/mL) solution: then add 7.0 mL of methanol.

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11.17.4. Transfer a portion of the sample to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the sample for re-injection or dilution.

11.18. Instrument Analysis

Suggested operating conditions are listed in Tables 1-7 for the Waters and SCIEX LCMS systems:

Table 1 - Recommended Instrument Operating Conditions				
HPLC Conditions (***)***				
Column (Column temp = C)				
Mobile Phase Composition	A =		B =	
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate - mL/min**
	Maximum pressure limit = 5,000 psi			
Injection Size	(fixed amount throughout the sequence). If non-concentrated extract then use .			
Run Time	~			
Mass Spectrometer Interface Settings (***)***				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.			
Ion Spray Voltage (kV)				
Entrance Potential (V)				
Declustering Potential (V)				
Desolvation Temp				
Curtain Gas				
Collision Gas				

Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ( )								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFBA	Native analyte	212.9 > 169						
13C4-PFBA	IDA	217 > 172						
PFBS	Native analyte	298.9 > 80						
PFBS_2	Native analyte	298.9 > 99						

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Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ([redacted])								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
13C3-PFBS	IDA	301.9 > 83	0.011	[redacted]				
PFPeA	Native analyte	262.9 > 219	0.011					
13C5-PFPeA	IDA	267.9 > 223	0.011					
4:2 FTS	Native analyte	327 > 307	0.011					
M2-4:2FTS	IDA or Reverse Surrogate for TOP	329 > 81	0.011					
PFHxA	Native analyte	313 > 269	0.011					
PFHxA_2	Native analyte	313 > 119	0.011					
13C2-PFHxA	IDA	315 > 270	0.011					
PFHpA	Native analyte	363 > 319	0.011					
PFHpA_2	Native analyte	363 > 169	0.011					
13C4-PFHpA	IDA	367 > 322	0.011					
PFPeS	Native analyte	349 > 80	0.011					
PFPeS_2	Native analyte	349 > 99	0.011					
PFHxS	Native analyte	399 > 80	0.011					
PFHxS_2	Native analyte	399 > 99	0.011					
18O2-PFHxS	IDA	403 > 84	0.011					
6:2 FTS	Native analyte	427 > 407	0.011					
M2-6:2FTS	IDA	429 > 81	0.011					
PFOA	Native analyte	413 > 369	0.011					
PFOA_2	Native analyte	413 > 169	0.011					
13C4-PFOA	IDA	417 > 372	0.011					
13C2-PFOA	IS	415 > 370	0.011					
PFHpS	Native analyte	449 > 80	0.011					
PFHpS_2	Native analyte	449 > 99	0.011					
PFNA	Native analyte	463 > 419	0.011					
PFNA_2	Native analyte	463 > 169	0.011					
13C5-PFNA	IDA	468 > 423	0.011					
PFOS	Native analyte	499 > 80	0.011					
PFOS_2	Native analyte	499 > 99	0.011					
PFNS	Native analyte	549 > 80	0.011					
PFNS_2	Native analyte	549 > 99	0.011					
PFDoS	Native analyte	699 > 80	0.011					
PFDoS_2	Native analyte	699 >99	0.011					
13C4-PFOS	IDA	503 > 80	0.011					
PFDA	Native analyte	513 > 469	0.011					
PFDA_2	Native analyte	513 > 169	0.011					
13C2-PFDA	IDA	515 > 470	0.011					
8:2 FTS	Native analyte	527 > 507	0.011					
10:2 FTS	Native analyte	627 > 607	0.011					

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Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ([redacted])								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
M2-8:2FTS	IDA	529 > 81	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFOSA	Native analyte	498 > 78	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C8-PFOSA	IDA	506 > 78	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
N-MeFOSAA	Native analyte	570 > 419	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
d3-MeFOSAA	IDA	573 > 419	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFDS	Native analyte	599 > 80	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFDS_2	Native analyte	599 > 99	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFUdA	Native analyte	563 > 519	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFUdA_2	Native analyte	563 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFUdA	IDA	565 > 520	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
N-EtFOSAA	Native analyte	584 > 419	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
d5-EtFOSAA	IDA	589 > 419	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFDoA	Native analyte	613 > 569	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFDoA_2	Native analyte	613 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFDoA	IDA	615 > 570	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFTrDA	Native analyte	663 > 619	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFTrDA_2	Native analyte	663 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFTeDA	Native analyte	713 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFTeDA_2	Native analyte	713 > 219	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFTeDA	IDA	715 > 670	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFHxDA	Native analyte	813 > 769	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFHxDA_2	Native analyte	813 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFHxDA	IDA	815 > 770	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFODA	Native analyte	913 > 869	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
PFODA_2	Native analyte	913 > 169	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 3 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ([redacted]) for Fluorinated Replacement Chemicals								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
HFPO-DA	Native analyte	329.1 > 285	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C3-HFPO-DA	IDA	332.1 > 287	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
9Cl-PF3ONS (F53B major)	Native analyte	531 > 351	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

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Table 3 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ([redacted]) for Fluorinated Replacement Chemicals								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
11Cl-PF3OUdS (F53B minor)	Native analyte	631 > 451	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
Dona	Native analyte	377 > 251	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
Dona_2	Native analyte	377 > 85	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 4 - Retention Times & Quantitation ([redacted])				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	[redacted]	13C4-PFBA	[redacted]	Isotope Dilution
PFPeA	[redacted]	13C5-PFPeA	[redacted]	Isotope Dilution
PFBS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHxA	[redacted]	13C2-PFHxA	[redacted]	Isotope Dilution
PFPeS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHpA	[redacted]	13C4-PFHpA	[redacted]	Isotope Dilution
PFHxS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFOA	[redacted]	13C4-PFOA	[redacted]	Isotope Dilution
PFHpS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNA	[redacted]	13C5-PFNA	[redacted]	Isotope Dilution
PFOS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFDA	[redacted]	13C2-PFDA	[redacted]	Isotope Dilution
FOSA	[redacted]	13C8-FOSA	[redacted]	Isotope Dilution
PFDS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFUdA	[redacted]	13C2-PFUdA	[redacted]	Isotope Dilution
PFDoA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFTrDA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFDoS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFTeDA	[redacted]	13C2-PFTeDA	[redacted]	Isotope Dilution
PFHxDA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution
PFODA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution
EtFOSAA	[redacted]	d5-EtFOSAA	[redacted]	Isotope Dilution
MeFOSAA	[redacted]	d3-MeFOSAA	[redacted]	Isotope Dilution
4:2 FTS	[redacted]	M2-4:2 FTS (If TOP then 13C-PFBS)	[redacted]	Isotope Dilution
6:2FTS	[redacted]	M2-6:2FTS	[redacted]	Isotope Dilution
8:2FTS	[redacted]	M2-8:2FTS	[redacted]	Isotope Dilution
HFPO-DA	[redacted]	13C3-HFPO-DA	[redacted]	Isotope Dilution

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Table 4 - Retention Times & Quantitation ( )				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
9Cl-PF3ONS (F53B major)		13C4-PFOS		Isotope Dilution
11Cl-PF3OUdS (F53B minor)		13C4-PFOS		Isotope Dilution
Dona		13C4-PFOS		Isotope Dilution
10:2 FTS		M2-8:2 FTS		Isotope Dilution

Table 5 - Recommended Instrument Operating Conditions					
HPLC Conditions ( )					
Column (Column temp = °C)	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm				
Mobile Phase Composition	A =			B =	
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate - mL/min.**
	Maximum pressure limit = 15,000 psi				
Injection Size	µL (fixed amount throughout the sequence)				
Run Time	~				
Mass Spectrometer Interface Settings (Quattro Premier XE)					
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.				
Capillary (kV)					
Cone (V)					
Extractor (V)					
Source Temp					
Desolvation Temp					
Cone Gas (nitrogen) Flow					
Desolvation Gas (nitrogen) Flow					

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Table 6 - Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings ([redacted])						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Native analyte	213 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFBA	IDA	217 > 172	[redacted]	[redacted]	[redacted]	[redacted]
PFPeA	Native analyte	263 > 219	[redacted]	[redacted]	[redacted]	[redacted]
13C5-PFPeA	IDA	268 > 223	[redacted]	[redacted]	[redacted]	[redacted]
PFBS	Native analyte	299 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFBS_2	Native analyte	299 > 99	[redacted]	[redacted]	[redacted]	[redacted]
13C3-PFBS	IDA	302 > 83	[redacted]	[redacted]	[redacted]	[redacted]
PFHxA	Native analyte	313 > 269	[redacted]	[redacted]	[redacted]	[redacted]
PFHxA_2	Native analyte	313 > 119	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFHxA	IDA	315 > 270	[redacted]	[redacted]	[redacted]	[redacted]
PFHpA	Native analyte	363 > 319	[redacted]	[redacted]	[redacted]	[redacted]
PFHpA_2	Native analyte	363 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFHpA	IDA	367 > 322	[redacted]	[redacted]	[redacted]	[redacted]
PFHxS	Native analyte	399 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFHxS_2	Native analyte	339 > 99	[redacted]	[redacted]	[redacted]	[redacted]
18O2-PFHxS	IDA	403 > 84	[redacted]	[redacted]	[redacted]	[redacted]
PFOA	Native analyte	413 > 369	[redacted]	[redacted]	[redacted]	[redacted]
PFOA_2	Native analyte	413 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFOA	IS	415 > 370	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFOA	IDA	417 > 372	[redacted]	[redacted]	[redacted]	[redacted]
PFHpS	Native analyte	449 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFHpS_2	Native analyte	449 > 99	[redacted]	[redacted]	[redacted]	[redacted]
PFNA	Native analyte	463 > 419	[redacted]	[redacted]	[redacted]	[redacted]
PFNA_2	Native analyte	463 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C5-PFNA	IDA	468 > 423	[redacted]	[redacted]	[redacted]	[redacted]
PFOS	Native analyte	499 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFOS_2	Native analyte	499 > 99	[redacted]	[redacted]	[redacted]	[redacted]
PFNS	Native analyte	549 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFNS_2	Native analyte	549 > 99	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFOS	IDA	503 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFDA	Native analyte	513 > 469	[redacted]	[redacted]	[redacted]	[redacted]
PFDA_2	Native analyte	513 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFDA	IDA	515 > 470	[redacted]	[redacted]	[redacted]	[redacted]
PFUdA	Native analyte	563 > 519	[redacted]	[redacted]	[redacted]	[redacted]
PFUdA_2	Native analyte	563 > 169	[redacted]	[redacted]	[redacted]	[redacted]

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Table 6 - Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings ([redacted])						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
13C2-PFUdA	IDA	565 > 520	[redacted]	[redacted]	[redacted]	[redacted]
PFDS	Native analyte	599 > 80	[redacted]	[redacted]	[redacted]	[redacted]
PFDS_2	Native analyte	559 > 99	[redacted]	[redacted]	[redacted]	[redacted]
FOSA	Native analyte	498 > 78	[redacted]	[redacted]	[redacted]	[redacted]
13C8-FOSA	IDA	506 > 78	[redacted]	[redacted]	[redacted]	[redacted]
PFDoA	Native analyte	613 > 569	[redacted]	[redacted]	[redacted]	[redacted]
PFDoA_2	Native analyte	613 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFDoA	IDA	615 > 570	[redacted]	[redacted]	[redacted]	[redacted]
PFTrDA	Native analyte	663 > 619	[redacted]	[redacted]	[redacted]	[redacted]
PFTrDA_2	Native analyte	663 > 169	[redacted]	[redacted]	[redacted]	[redacted]
PFTeDA	Native analyte	713 > 169	[redacted]	[redacted]	[redacted]	[redacted]
PFTeDA_2	Native analyte	713 > 219	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFTeDA	IDA	715 > 670	[redacted]	[redacted]	[redacted]	[redacted]
PFHxDA	Native analyte	813 > 769	[redacted]	[redacted]	[redacted]	[redacted]
PFHxDA_2	Native analyte	813 > 169	[redacted]	[redacted]	[redacted]	[redacted]
PFODA	Native analyte	913 > 869	[redacted]	[redacted]	[redacted]	[redacted]
PFODA_2	Native analyte	913 > 169	[redacted]	[redacted]	[redacted]	[redacted]
13C2-PFHxDA	IDA	815 > 770	[redacted]	[redacted]	[redacted]	[redacted]
EtFOSAA	Native analyte	584 > 419	[redacted]	[redacted]	[redacted]	[redacted]
d5-EtFOSAA	IDA	589 > 419	[redacted]	[redacted]	[redacted]	[redacted]
MeFOSAA	Native analyte	570 > 419	[redacted]	[redacted]	[redacted]	[redacted]
d3-MeFOSAA	IDA	573 > 419	[redacted]	[redacted]	[redacted]	[redacted]
4:2FTS	Native analyte	327 > 307	[redacted]	[redacted]	[redacted]	[redacted]
M2-4:2FTS	IDA or Reverse Surrogate for TOP	329 > 81	[redacted]	[redacted]	[redacted]	[redacted]
6:2FTS	Native analyte	427 > 407	[redacted]	[redacted]	[redacted]	[redacted]
M2-6:2FTS	IDA	429 > 81	[redacted]	[redacted]	[redacted]	[redacted]
8:2FTS	Native analyte	527 > 507	[redacted]	[redacted]	[redacted]	[redacted]
M2-8:2FTS	IDA	529 > 81	[redacted]	[redacted]	[redacted]	[redacted]

Table 7 - Recommended Instrument Operating Conditions				
Retention Times & Quantitation ([redacted])				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	[redacted]	13C4-PFBA	[redacted]	Isotope Dilution

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Table 7 - Recommended Instrument Operating Conditions				
Retention Times & Quantitation ([redacted])				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFPeA	[redacted]	13C5-PFPeA	[redacted]	Isotope Dilution
PFBS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHxA	[redacted]	13C2-PFHxA	[redacted]	Isotope Dilution
PFPeS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFHpA	[redacted]	13C4-PFHpA	[redacted]	Isotope Dilution
PFHxS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFOA	[redacted]	13C4-PFOA	[redacted]	Isotope Dilution
PFHpS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNA	[redacted]	13C5-PFNA	[redacted]	Isotope Dilution
PFOS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFDA	[redacted]	13C2-PFDA	[redacted]	Isotope Dilution
FOSA	[redacted]	13C8-FOSA	[redacted]	Isotope Dilution
PFDS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFUdA	[redacted]	13C2-PFUdA	[redacted]	Isotope Dilution
PFDoA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFTrDA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFTeDA	[redacted]	13C2-PFTeDA	[redacted]	Isotope Dilution
PFHxDA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution
PFODA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution
EtFOSAA	[redacted]	d5-EtFOSAA	[redacted]	Isotope Dilution
MeFOSAA	[redacted]	d3-MeFOSAA	[redacted]	Isotope Dilution
4:2FTS	[redacted]	M2-4:2 FTS (If TOP then 13C-PFBS)	[redacted]	Isotope Dilution
6:2FTS	[redacted]	M2-6:2FTS	[redacted]	Isotope Dilution
8:2FTS	[redacted]	M2-8:2FTS	[redacted]	Isotope Dilution

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11.18.1. Post Spike Sample Analysis for AFFF samples

11.18.1.1. This section only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <LOQ (RL) for any analyte.

11.18.1.2. Spike aliquots of the sample at the final dilution reported for the sample with all analytes that have reported of <LOQ in the final dilution. The spike must be at the LOQ concentration to be reported with the sample (the < LOQ value).

11.18.1.3. When analyte concentrations are calculated as <LOQ, the spike must recover within 70-130% of its true value.

11.18.1.4. It the recovery does not meet this criteria, the sample, sample duplicate and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.

11.18.2. Tune and calibrate the instrument as described in Section 10.

11.18.3. A typical run sequence is as follows:

- Rinse Blank (RB, not linked to anything)
- Start ICAL with CCVL but called IC in TALS (starts the 12 hour clock or time 0:00)
- Rest of ICAL
- ICB: link to midpoint of ICAL and samples
- ICV: link to midpoint of ICAL and samples (If ICAL good)
- CCB: link to midpoint of ICAL and samples
- PFOA RT marker
- Rinse Blank (RB, not linked to anything)
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- 10 more samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- Etc.
- CCVL (within 12 hours from CCVL in ICAL, can be the ending CCV and starts 12 hours all over again): if this occurs link to the midpoint of the ICAL/toggle it as opening/closing CCV.
- CCV: link to midpoint of ICAL
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- If no ICAL run that day
- CCB: link to CCVIS
- CCVL (starts 12 hour clock): link to CCVIS
- CCVIS: link to midpoint of ICAL
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- Etc.

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- If going over 12 hours in the sequence: CCVL (within 12 hours from CCVL at item 2 above, can be the ending CCV and starts 12 hours all over again): if this occurs link to the CCVIS /toggle as opening and closing CCV.
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFAS with labeled standards should 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 IDA RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 3

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

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12.5. Water Sample Result Calculation:

Equation 5

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex}	=	Concentration measured in sample extract (ng/mL)
V_t	=	Volume of total extract (mL)
V_o	=	Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

Equation 6

$$\text{Concentration, } ng/g = \frac{C_{ex} V_t}{W_s D}$$

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)

12.7. IDA Recovery Calculation:

Equation 7

$$\%\,\text{Recovery} = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X 100$$

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

12.8. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

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13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006 and policy WS-PQA-003. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

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

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15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids saturated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel flammable solvent drum to the waste collection area for shipment.

15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full, to no less than six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, “Determination of Perfluorinated Surfactants in Surface Water Samples by

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Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and 19FNMR," Analytical Chemistry 2001, 73, 2200-2206.

16.2. John Giesy et al., "Accumulation of Perfluorooctane Sulfonate in Marine Mammals", Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.

16.3. U.S. EPA, "Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method", EPA 712-C-95-174, August 1995.

16.4. STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.

16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.

16.6. 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.

16.7. 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.

16.8. 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

16.9. Erika F. Houtz and David L. Sedlak, "Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff," Environmental Science and Technology 46, no. 17 (2012): 9342-49.

17. METHOD MODIFICATIONS

17.1. Modifications from Method 537 are detailed below:

17.1.1. Water sample containers are not preserved with Trizma.

17.1.2. The method has been modified to address soil/solid matrices. The extraction

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holding time is set at 14 days.

17.1.3. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.

17.1.4. The reporting limits differ as they are all set at one consistent value.

17.1.5. Calibration levels differ from the referenced method.

17.1.6. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.

17.1.7. There is no symmetry requirement.

17.1.8. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of isotope dilution quantitation.

17.1.9. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol:water.

17.1.10. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.

17.1.11. Samples are not checked for residual chlorine or pH.

17.1.12. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

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18. ATTACHMENTS

18.1. Attachment 1 - Analysis of Perfluorinated Compounds (PFAS) in Water via In Line Solid Phase Extraction (SPE).

19. REVISION HISTORY

Revisions to Attachment 1 are documented in the attachment.

Revisions prior to 05/01/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Revision 3.5, Effective 02/27/2019

19.1.1. Added Section 11.3.6, “After the entire sample has been loaded onto the column, rinse the sample bottle with two 5 mL aliquots of reagent water and pour onto the column reservoir.”

19.1.2. Editorial changes.

19.2. WS-LC-0025, Revision 3.4, Effective 02/13/2019

19.2.1. Section 6.4 added, “The average weight of the HDPE bottles with HDPE screw caps are calibrated once a year. The calibration is performed by weighing 10 bottles with caps and dividing by 10 to get the average weight. The average weight is used in section (11.3.5.1.d).”

19.2.2. Section 7.4.1 revised, “an” to “every” and removed “or when a new column is installed”.

19.2.3. Add Section 7.4.1.1, “Attach this document to the ICV from the associated ICAL by scanning the document and associating it to the file as a document type of High Res MS Tune in TALS. Use the following naming convention: “_ZbatchnumberTPFOA”.”

19.2.4. Added Section 8.2.1, “Projects performed for the state of New Jersey have an analytical holding time 28 days from the extraction date.”

19.2.5. Added Section 8.2.2, “For projects performed for the state of New Jersey a field reagent blank (FRB) must be collected with each sample set. Acceptance limits are <RL for each analyte.”

19.2.6. Added Section 9.4.1, “Projects performed for the state of New Jersey: LCS (mid and high spike) recovery limits are 70-130%. Low level LCS recovery limits are 50-150%. The spike level must rotate between low, medium and high.”

19.2.7. Added Section 9.5.1, “Projects performed for the state of New Jersey: MS/MSD (mid and high spike) recovery limits are 70-130%. Low level MS/MSD recovery limits are 50-150%. The spike level must rotate between low, medium and high.”

19.2.8. Added Section 9.10, “TOP Oxidation Efficiency” and its associated subsections.

19.2.9. Added Section 9.11, “Ion Ratio” and associated subsections.

19.2.10. Added Section 10.8.2.6, “Projects performed for the state of New Jersey: MS/MSD (mid and high spike) recovery limits are 70-130%. Low level MS/MSD recovery limits are 50-150%. The spike level must rotate between low, medium and high.”

19.2.11. Section 10.11.3 added, “and the state of New Jersey”.

19.2.12. Added Section 10.12.5, “Projects performed for the state of New Jersey: All

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analyte concentrations in the CCV must be within + 30% of their true value. All analyte concentrations in the low level CCV must be within + 50% of their true value."

19.2.13. Added Section 11.3.5.1, "If the SPE column should plug (flow rate <1 drop per minute) prior to the entire content of the sample container passing through the column do the following:" and its associated subsections.

19.2.14. Sections 11.14.15.8 and 11.15.27.8 removed, "with a flow rate of 1 mL/minute".

19.2.15. Section 11.18.3 removed, "(as needed)" from the PFOA RT marker.

19.2.16. Throughout SOP revised, "1 mL/minute" to "3-5 drops per second".

19.2.17. Editorial changes.

19.3. WS-LC-0025, Revision 3.3, Effective 12/03/2018

19.3.1. Added Section 6.9, "[redacted]."

19.3.2. Tables 2 and 6 revised comment for M2-4:2 FTS to, "IDA or Reverse Surrogate for TOP".

19.3.3. Tables 4 and 7 revised header from "IS Analog" to "IDA Analog", and revised "4:2 FTS" entry to "M2-4:2 FTS (If TOP then 13C-PFBS)".

19.3.4. Editorial changes.

19.4. WS-LC-0025, Revision 3.2, Effective 08/20/2018

19.4.1. Section 1 added, "1H,1H,2H,2H-perfluorododecane sulfonate" and "Perfluoro-1-dodecansulfonic acid" entries to table.

19.4.2. Section 1.2 revised table entry for "Adona" to "Dona".

19.4.3. Section 7.4 added, "PFDoS" and "10:2 FTS" entries to table.

19.4.4. Section 7.4 revised, "Adona" entry to "Dona".

19.4.5. Table 2 added, "PFDoS", "PFDoS_2", and "10:2 FTS" entries to table.

19.4.6. Table 3 revised, "Adona" and "Adona_2" entries to "Dona" and "Dona_2".

19.4.7. Table 4 added, "PFDoS" and "10:2 FTS" entries to table.

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19.4.8. Table 4 revised, “Adona entry to “Dona”.

19.4.9. Editorial changes.

19.5. WS-LC-0025, Revision 3.1, Effective 06/21/2018

19.5.1. Section 11.2.1 revised to, “Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action. Decanting or filtering of the sample can lead to a low bias.”

19.5.2. Editorial changes.

19.6. WS-LC-0025, Revision 3.0, Effective 04/13/2018

19.6.1. Section 1.1 updated table with PFPeS and PFNS analytes.

19.6.2. Added Section 2.2, which details the analytes that can be covered by the method under special request.

19.6.3. Added Section 3.13, “AFFF: Aqueous Film Forming Foam”.

19.6.4. Section 6.19 added, “Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use” to table.

19.6.5. Section 7.1.2 added, “[redacted] The resultant solution is filtered through a 0.22um filter before use. This solution has volatile components, thus it should be replaced every 7 days or sooner.”

19.6.6. Section 7.1.3 added, “[redacted].”

19.6.7. Section 7.1.8 added, “[redacted].”

19.6.8. Section 7.1.11 added, “Prepared by diluting 400mL of 1N NaOH into 3.6L of water for a total volume of 4L.”

19.6.9. Section 7.4 updated table with PFPeS and PFNS analytes.

19.6.10. Section 7.4, added table to detail ICAL for Fluorinated Replacement Compounds.

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19.6.11. Added Section 8.1.1, “Water samples collected from a known chlorinated source should be preserved with Trizma.”

19.6.12. Added Section 9.9.3, “If the IS does not meet criteria, re-analyze the extract. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration.”

19.6.13. Added Section 11.14.6, “Add 2g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.”

19.6.14. Removed Section 11.14.8, “Add 2g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.”

19.6.15. Added Section 11.14.9, “Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.”

19.6.16. Added Section 11.5 and associated subsections, which detail the “TOPS (Total Oxidizable Precursor) Assay for Soil Sample”.

19.6.17. Section 11.8 updated Table labeling, added PFPeS and PFNS analytes throughout Tables where applicable, and updated Table 7 to reflect current retention times and quantitation.

19.6.18. Section 11.8 added Table 6, “Recommended Instrument Operating Conditions Mass Spectrometer Scan Settings ([redacted]) for Fluorinated Replacement Chemicals”

19.6.19. Section 11.18.3 removed outdated run sequence and replaced with current run sequence.

19.6.20. Editorial changes.

19.7. WS-LC-0025, Revision 2.9, Effective 11/22/2017

19.7.1. Section 1.2, table updated to reflect ranges after removing MeFOSA and EtFOSA from the SOP in the previous revision.

19.7.2. Section 9.3.6, last sentence changed to read, “Reprepare and reanalyze all field and QC samples associated with the contaminated method blank.”

19.7.3. Section 9.7, first sentence changed to read, “Initial calibration verification (ICV) – A second source standard is analyzed with the initial calibration curve.

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19.7.4. Section 1.3.1 revised to read, “Once the optimal mass assignments (within ±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion. For QSM work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met.”

19.7.5. Editorial changes.

19.8. WS-LC-0025, Revision 2.8, Effective 11/06/2017

19.8.1. Revised Section 4.5 to “Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PFBS, EtFOSAA, and MeFOSAA based upon the literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolved or not, but usually with a deflection point resolved during peak integration. The later of these peaks match the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

At this time only PFOS, PFOA and PFHxS 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.”

19.8.2. Sections 4.8 and 7.2.1.1, corrected the in-sample contributions to 0.30 ng/L and 0.015 ug/kg.

19.8.3. Removed Section 7.1.14, “Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap.” Reagent was added incorrectly.

19.8.4. Section 7.2.4, corrected the factor to 0.956 from 1.046.

19.8.5. Added Section 7.4.1, “A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and

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branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard This technical (qualitative) grade PFOA standard is analyzed initially, after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters."

19.8.6. Section 9.7, added "Rerun the initial calibration" as the last bullet item.

19.8.7. Added Section 10.3.1, "The first level standard from the initial calibration curve is used to evaluate the tune criteria. The instrument mass windows are set at ±0.5 amu; therefore, detection of the analyte serves as verification that the assigned mass is within ±0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion.

19.8.8. Section 10.10.1, appended "containing both IDA and IS" to the end of the paragraph.

19.8.9. Sections 11.6.3 and 11.12.2.3, changed "78:22 methanol:water" to "methanol".

19.8.10. Sections 1.1 and 7.4, removed EtFOSA and MeFOSA from tables due to low volume of requests for those analytes.

19.8.11. Removed Section 2.2.1, "Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSA and MeFOSA are requested."

19.8.12. Removed EtFOSA/MeFOSA specific comments in various sections throughout the document.

19.8.13. Section 7.4 Note added, "The concentration of the calibration solutions for non-concentrated extracts is 1/20th the levels indicated above."

19.8.14. Section 7.9, changed 1000 ng/mL to 250 ng/mL and replaced final sentence with "The internal standard solution used for the non-concentrated extracts is at a concentration of 50 ng/mL."

19.8.15. Removed Section 11.2.8, "If EtFOSA and/or MeFOSA are requested, add 100uL of IS and then adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. QC samples, LCS, MS, and MSD will require concentration via nitrogen to adjust the FV to 5.0 mL. Vortex each sample. Then, transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution."

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19.8.16. Added Section 11.5.4, “Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This is required for all DoD/DOE extracts.”

19.8.17. Added Section 11.7.1.1, “Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step.”

19.8.18. Inserted Section 11.8.1.1, “Projects performed under the auspices of the DoD/DOE must have the entire sample homogenized prior to subsampling in accordance with QSM 5.1 criteria.”

19.8.19. Section 11.11.4, added “(Graphitized Carbon Cleanup) as needed. This is required for all DoD/DOE extracts.”

19.8.20. Section 11.14.6, added “Spike all “Pre” and “Post” samples with 25uL of the reverse surrogate solution (Section 7.8).”

19.8.21. Section 11.15.2, revised to read, “Cleanup with graphitized carbon will be applied to all samples as needed but is required for all DoD/DOE extracts.”

19.8.22. Added Section 11.15.2.5, “Proceed to Section 11.6, 11.7, or 11.12 as applicable.”

19.8.23. Removed Sections 11.15.3 through 11.15.6.

19.8.24. Added Section 11.16, “AFFF Sample Preparation”.

19.8.25. Section 11.17, removed EtFOSA, MeFOSA, d5-EtFOSA, and d3MeFOSA from all tables.

19.8.26. Section 11.17, changed masses for M2-4:2FTS, M2-6:2FTS, and M2-8:2FTS. Initially assigned daughter masses were bleeding through from the native analog.

19.8.27. Section 11.17, all tables on MS Interface Mode Line, added “Minimum of 10 scans/peak.”

19.8.28. Added Section 11.17.1, “Post Spike Sample Analysis for AFFF Samples”.

19.8.29. Added Section 11.8.4.1 “Spike non-concentrated samples at 0.5 mL of LCS/Matrix Spike Solution.”

19.8.30. Added Section 11.8.5.1, “Spike non-concentrated samples at 0.5 mL of IDA PFC Solution.”

19.8.31. Editorial changes.

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19.9. WS-LC-0025, Revision 2.7, Effective 09/20/2017

19.9.1. Section 1.1 table, added 1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*.

19.9.2. Section 1.1, removed "Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7)."

19.9.3. Section 1.2 and 11.8.2, updated tissue extracted mass and RL.

19.9.4. Section 2.5, removed "and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve."

19.9.5. Added Section 6.6, "Extract concentrator or nitrogen manifold with water bath heating to 50-55°C".

19.9.6. Added Section 7.1.14, "Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap."

19.9.7. Section 7.2.1.1, revised "roughly 0.15 pg/L" to "roughly 0.15 ng/L".

19.9.8. Section 7.4 table, added:

4:2 FTS	0.5	1.0	2.0	20	50	200	400

19.9.9. Section 7.4 table, revised Labeled Isotope Dilution Analytes (IDA) Section.

19.9.10. Section 7.4 table, added:

Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

19.9.11. Section 7.4, removed "FOSAA may be added to the mix and are added at the same concentration as FOSA."

19.9.12. Added Section 7.9, "Internal Standard Solution, 1000 ng/mL. The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all extracts prior to analysis. Non-concentrated extracts are fortified with a 5X dilution of this solution."

19.9.13. Section 8.1, changed "250 mL" to "8 oz."

19.9.14. Added Sections 9.3.6, 9.8.2.3, 10.10.4, 10.8.2.5, 10.11.3, and 10.12.4 to address DOD QSM 5.1 Table B-15 criteria.

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19.9.15. Added Section 9.9, “Internal Standard.”

19.9.16. Updated all tables to indicate target analyte quantitation via isotope dilution. Internal standard quantitation is only used to quantitate the IDA recoveries.

19.9.17. Added Section 10.8.2.4, 10.12.2, and 10.12.2.1 to incorporate IS criteria into calibrations.

19.9.18. Section 11.2.1, “Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.”

19.9.19. Added Section 11.2.3.1, “Alternatively, weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume.”

19.9.20. Added Section 11.5.3, “Note: If the extracts will not be concentrated elute extract with a total of 8 mL of [redacted].”

19.9.21. Added Section 11.6.2.3, “Add 300 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer.”

19.9.22. Added Section 11.6.2.4, “Add 100 uL of Internal Standard (IS) solution to each extract and vortex to mix.”

19.9.23. Added Section 11.7, “Final volume for non-concentrated extract”.

19.9.24. Revised Section 11.11, “SPE Elution of Solid Extracts”.

19.9.25. Revised Section 11.12, “Extract Concentration for Solid Samples”.

19.9.26. Removed Section 12.8, “If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)”

19.9.27. Removed Section 13.4 – it was a copy of Section 13.2.

19.9.28. Various revisions to fulfill requirements based on DOD/DOE QSM 5.1.

19.9.29. Editorial changes.

19.10. WS-LC-0025, Revision 2.6, Effective 08/15/2017

19.10.1. Section 7.4, added MPFBS, MPFTeDA, and MPFHxDA to the table.

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19.10.2. Section 11.15, added 13C-PFBS to the Recommended Instrument Operating Conditions table for [redacted].

19.10.3. Section 11.15 Recommended Instrument Operating Conditions table, changed the mass transitions for native PFTeDA from 713 > 669 (quant) and 713 > 169 (qualifier) to 713 > 169 (quant) and 713 > 219 (qualifier).

19.10.4. Editorial changes.

19.11. WS-LC-0025, Revision 2.5, Effective 07/10/2017

19.11.1. Revised Section 11.6.1 to read "Prior to concentrating each sample, add 100 uL of water."

19.11.2. Revised Section 11.6.2 to read "Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1 This blow down must take a minimum of 3.5 hours.

11.6.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated."

19.11.3. Revised Section 11.6.3 to read "Add 400 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

19.11.4. Revised Section 11.11.1 to read "Prior to concentrating each sample, add 200 uL of water."

19.11.5. Revised Section 11.11.2 to read "Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains."

11.11.2.1 This blow down must take a minimum of 3.5 hours.

11.11.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated."

19.11.6. Revised Section 11.11.3 to read "Add 800 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples via in line solid phase extraction (SPE) for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a [redacted].

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1

1.2. 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	1.0 mL	2.0 ng/L	2 to 200 ng/L

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2. SUMMARY OF METHOD

2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using [redacted].

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the in line SPE analysis.

6.1. 2 mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent.

6.2. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.

6.3. Eppendorf 1000 uL epTIPS, part no. 022491954 or equivalent.

6.4. Eppendorf 200 uL epTIPS, part no. 022491938 or equivalent.

6.5. 50 mL graduated plastic centrifuge tubes, SCP Science DigiTUBES part no. 010-500-263 or equivalent.

6.6. 1000 uL Pipette: Eppendorf Research Plus.

6.7. 100 uL Pipette: Rainin EDP3-Plus.

6.8. 250 mL HDPE bottles with PPE screw caps, ESS part no. 0250-1902-QC or equivalent.

6.9. Analytical columns

6.9.1. [redacted], or equivalent.

6.9.2. PFAS Isolator column, [redacted] or equivalent.

6.10. [redacted] Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.

6.11. [redacted] HPLC equipped with [redacted] pumps and [redacted] degassing unit or equivalent.

7. REAGENTS AND STANDARDS

Refer to Section 7 of the main body of this SOP for reagents and standards, other than those listed below specific to the in line SPE analysis.

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

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7.1.1. Ammonium acetate, Fisher Optima LCMS grade (20 mM in water), part no. A114-50, or equivalent.

7.1.2. Methanol, Baker HPLC grade, part no. 9093-03.

7.1.3. Water, Nanopure or Millipore or Fisher Optima LCMS grade, part no. W6-4, must be free of interference and target analytes.

7.2. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of the stock solutions (Section 7.2 of the main body of this SOP) in 40:60 methanol:water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.3. Initial Calibration (ICAL) Levels (ng/L)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
Perfluoroalkylcarboxylic acids (PFCAs)								
PFHpA	1.0	2.0	5.0	10	20	50	100	200
PFOA	1.0	2.0	5.0	10	20	50	100	200
PFNA	1.0	2.0	5.0	10	20	50	100	200
Perfluorinated sulfonic acids (PFSAs)								
PFBS	1.0	2.0	5.0	10	20	50	100	200
PFHxS	1.0	2.0	5.0	10	20	50	100	200
PFOS	1.0	2.0	5.0	10	20	50	100	200
Labeled Isotope Dilution Analytes (IDA)								
13C4-PFHpA	50	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50	50
13C3-PFBS	50	50	50	50	50	50	50	50

Note: *The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.*

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

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The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each at 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C. Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

9.1. If potable water samples from the state of New York (NY) are analyzed via this method the control limits for LCS and IDA for PFOS and PFOA recoveries are 70-130%. If these limits are not met, refer to Section 9 of the main body of this SOP for corrective action.

9.2. If POST (treatment) samples have positive detections, review the associated PRE and MID (treatment) samples for similar detections. Re-preparation and re-analysis may be needed.

9.3. If PFBS is detected in the method blank greater than the RL, evaluate data for impact. PFBS is a known laboratory artifact. Re-preparation and re-analysis may be needed.

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10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the in line SPE analysis.

11.1. Water Sample Preparation

11.1.1. Visually inspect samples for the presence of settled and or suspended sediment/particulate. Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.

If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent).

Gravity of vacuum can be used to pass the sample through the filter. Prepare a filtration blank 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.

11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.

11.1.3. If requested, find the client assigned sample for MS/MSD.

11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.

11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

11.1.6. Be sure to "prepare" the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.

11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.

11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 40:60 methanol:water.

11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed in Tables 1A-1C below:

Table 1A - Routine Instrument Operating Conditions	
HPLC Conditions ([redacted])	
Column (Column temp = [redacted]°C)	[redacted]
Mobile Phase Composition	A = [redacted] B = [redacted]

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Table 1A - Routine Instrument Operating Conditions					
HPLC Conditions ([redacted] ***)***					
Gradient Program	**Time (min)**	**%A**	**%B**	**Curve**	**Flow Rate (mL/min)**
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	Maximum Pressure limit = 5,000 psi				
Injection Size	[redacted] (fixed amount throughout the sequence)				
Run Time	[redacted]				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.				
Ion Spray Voltage (kV)	[redacted]				
Entrance Potential (V)	[redacted]				
Declustering Potential (V)	[redacted]				
Desolvation Temp	[redacted]				
Curtain Gas (nitrogen) Flow	[redacted]				
Collision Gas (nitrogen) Flow	[redacted]				

Table 1B - Routine Instrument Operating Conditions						
Mass Spectrometer Scan Settings ([redacted] ***)***						
Compound	**Comments**	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**
PFBS	Perfluorobutanesulfonate	299 > 80	[redacted]	[redacted]	[redacted]	[redacted]
13C3-PFBS	IDA	302 > 83	[redacted]	[redacted]	[redacted]	[redacted]
PFHpA	Perfluoroheptanoic acid	363 > 319	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFHpA	IDA	367 > 322	[redacted]	[redacted]	[redacted]	[redacted]
PFHxS	Perfluorohexanesulfonate	399 > 80	[redacted]	[redacted]	[redacted]	[redacted]
18O2-PFHxS	IDA	403 > 84	[redacted]	[redacted]	[redacted]	[redacted]
PFOA	Perfluorooctanoic acid	413 > 369	[redacted]	[redacted]	[redacted]	[redacted]
13C4PFOA	IDA	417 > 372	[redacted]	[redacted]	[redacted]	[redacted]
PFNA	Perfluorononanoic acid	463 > 419	[redacted]	[redacted]	[redacted]	[redacted]
13C5-PFNA	IDA	468 > 423	[redacted]	[redacted]	[redacted]	[redacted]
PFOS	Perfluorooctanesulfonate	499 > 80	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFOS	IDA	503 > 80	[redacted]	[redacted]	[redacted]	[redacted]

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Table 1C					
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHpA	[redacted]	13C4-PFHpA	[redacted]	Isotope Dilution
PFHxS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFOA	[redacted]	13C4-PFOA	[redacted]	Isotope Dilution
PFNA	[redacted]	13C5-PFNA	[redacted]	Isotope Dilution
PFOS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

11.2.3. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- PFOA RT marker (as needed)
- Rinse Blank (RB, not linked to anything)
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

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12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

14. POLLUTION PREVENTION

Refer to Section 14 of the main body of this SOP for pollution prevention information.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for waste management information.

16. REFERENCES

Refer to Section 16 of the main body of this SOP for reference information.

17. METHOD MODIFICATIONS

17.1. Refer to Section 17 of the main body of this SOP for modifications from Method 537, except as detailed below:

17.1.1. Water samples are prepared at 1.0 mL, not 250 mL.

17.1.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 28 days for analysis.

17.1.3. The eluents and HPLC configuration differs. As a result the final extract is in 40:60 methanol:water.

18. ATTACHMENTS

There are no attachments to this Appendix.

19. REVISION HISTORY

Revisions prior to 04/10/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Attachment 1, Revision 3.5, Effective 02/27/2019

19.1.1. No changes to the attachment with this revision.

19.2. WS-LC-0025, Attachment 1, Revision 3.4, Effective 02/13/2019

19.2.1. Removed Section 3.6, “MPFOA: Perfluoro-n-[1,2,3,4-13C4]octanoic acid. Carbon-13 labeled PFOA”.

19.2.2. Removed Section 3.7, “MPFOS: Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid. Carbon-13 labeled PFOS”.

19.2.3. Section 7.2.3 removed, “MPFOS”.

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19.2.4. Section 7.3 removed, “PFCA and PFSA”.

19.2.5. Section 7.3 added “13C3-PFBS” entry to table.

19.2.6. Section 10.11.3 revised to, “Projects performed under the auspices of the DoD/DOE QSM (Version 5.1) and the state of New Jersey must meet these criteria for the ICV: Analyte concentrations must be within ±30% of their true values for all analytes, IDA and target.”

19.2.7. Table 1B, revised PFBS IDA from “18O2-PFHxS” to “13C3-PFBS” and updated entry values.

19.2.8. Table 1C, revised “IS Analog” to “IDA Analog”, revised the PFBS IDA from “18O2-PFHxS” to “13C3-PFBS”, and updated entry values.

19.2.9. Editorial changes.

19.3. WS-LC-0025, Attachment 1, Revision 3.3, Effective 12/03/2018

19.3.1. No changes to the attachment with this revision.

19.4. WS-LC-0025, Attachment 1, Revision 3.2, Effective 08/20/2018

19.4.1. No changes to the attachment with this revision.

19.5. WS-LC-0025, Attachment 1, Revision 3.1, Effective 06/21/2018

19.5.1. No changes to the attachment with this revision.

19.6. WS-LC-0025, Attachment 1, Revision 3.0, Effective 04/13/2018

19.6.1. Updated labeling and formatting of Tables 1A-1C.

19.6.2. Added section 11.2.3, detailing a typical run sequence.

19.7. WS-LC-0025, Attachment 1, Revision 2.9, Effective 11/27/2017

19.7.1. No changes to the attachment with this revision.

19.8. WS-LC-0025, Attachment 1, Revision 2.8, Effective 11/06/2017

19.8.1. Section 11.2.1, Routine Instrument Operating Conditions table ([redacted]), added “Minimum of 10 scans/peak”.

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19.9. WS-LC-0025, Attachment 1, Revision 2.7, Effective 09/22/2017

19.9.1. Section 6.5, removed "The 5 items above are to be maintained in the drawer labeled "Segregated Supplies for in line SPE Analysis" in the LC/MS instrument room."

19.9.2. Added Sections 9.1 – 9.3.

19.9.3. Updated Section 11.1.

19.9.4. Editorial changes.

19.10. WS-LC-0025 Attachment 1, Revision 2.6, Effective 08/11/2017

19.10.1. No revisions to this attachment.

19.11. WS-LC-0025 Attachment 1, Revision 2.5, Effective 07/10/2017

19.11.1. No revisions to this attachment.

19.12. WS-LC-0025 Attachment 1, Revision 2.4, Effective 04/25/2017

19.12.1. No revisions to this attachment.

19.13. WS-LC-0025 Attachment 1, Revision 2.3, Effective 04/10/2017

19.13.1. Changed all mentions of "direct aqueous injection (DAI)" to "in line solid phase extraction (SPE)."

19.13.2. Inserted Section 17.1, and changed formatting of the modifications to Method 537 to Section 17.2 and subheadings.

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