SENSIBLE SOLUTIONS

Corporate Office
465 South Main Street
PO Box 639
Brewer, Maine 04412
207.989.4824

www.ces-maine.com

CES INC

DRAFT FISH TISSUE FIELD SAMPLING WORK PLAN BERRY'S BROOK COAKLEY LANDFILL SUPERFUND SITE NORTH HAMPTON AND GREENLAND, NEW HAMPSHIRE

FOR

THE COAKLEY LANDFILL GROUP

1 Junkins Avenue
Portsmouth, New Hampshire

May 15, 2018
JN: 10424.018

Prepared by:
CES, Inc.
640 Main Street
Lewiston, Maine 04240
207.795.6009

SEMS Doc ID 100009512

Engineers ◆ Environmental Scientists ◆ Surveyors

TABLE OF CONTENTS

FISH TISSUE SAMPLING FIELD WORK PLAN
BERRY'S BROOK
COAKLEY LANDFILL SUPERFUND SITE
NORTH HAMPTON AND GREENLAND, NEW HAMPSHIRE

1.0 | INTRODUCTION

On behalf the Coakley Landfill Group (CLG), CES, Inc. (CES) has prepared this Fish Tissue Sampling Field Work Plan (Work Plan) to provide an approach and protocols for the collection and analysis of fish tissue from locations along Berry's Brook, which runs through portions of Portsmouth, Greenland, and Rye, New Hampshire. The intent of the sampling is to collect representative samples of fish tissue from portions of Berry's Brook located downstream of Coakley Landfill Superfund Site (the Site) and analyze the tissue samples for the presence of per- and polyfluoroalkyl substances (PFAS).

The sampling is being conducted by the CLG, at the request of the U. S. Environmental Protection Agency (EPA) and New Hampshire Department of Environmental Services (NHDES).

2.0 | PROJECT BACKGROUND AND DESCRIPTION

Berry's Brook is a small freshwater stream that originates in a large wetland complex located west of the Coakley Landfill and flows in roughly northeasterly direction toward the mouth of the Piscataqua River. Berry's Brook is approximately 5.5 miles in length from its source to the estuary beginning at Bracket Road in Portsmouth, New Hampshire. The brook crosses several roads including Lafayette Road (closest to the Site), Lang Road, Sagamore Road, and finally Brackett Road where it begins forming an estuary and mixes with salt water from the mouth of the Piscataqua River. The Brook is a warm-water brook, which may provide habitat for native warm water fish species such as bull head, bass, perch, and pickerel, among other species which may be caught and/or consumed by recreational anglers.

The New Hampshire Fish and Game Department (NHF&G) stocks the estuary portion of Berry's Brook twice per year with hatchery-raised brown trout. Historically the stocking program was part of a "put and take" fishery management strategy, however, in 2018, the strategy has been changed to a "catch and release" program. Stocking occurs twice per year (typically April and September) near the Brackett Road crossing. In 2015, a total of 4,499 yearling brown trout were stocked at the Brackett Road crossing, of these, 1,999 were stocked in the spring and 2,500 were stocked in the fall. It has not been determined if the stocked fish travel from the stocking location, upstream, beyond the Sagamore Road crossing (which is the most upstream location identified by NHFG, as having recreational fishing activity). Brown trout are also known to migrate to the ocean. Evidence of brown trout spawning (the presence of redds) has been reported in the Brackett Road crossing area. Recreational fishing activities have not been documented by NH F&G upstream of the stocking area, however there are four road crossing access points to the brook, from which such activities could occur. NHF&G records from 1989 through 2014 show that an overall average of 24 brown trout were caught by anglers per year from the Brackett Road area. Current records for recreational fishing activity/success were not available.

Surface water samples from Berry's Brook, collected by the New Hampshire Department of Environmental Service (NHDES) in 2016, and by the CLG in 2017 identified detectable concentrations of PFAS in the collected surface water samples. The samples were collected from locations along Berry's Brook, adjacent to the Site, and downstream to the Lafayette Road crossing which is approximately 1.4 miles from the Site. Sampling for the presence of PFAS, in the remaining portion of Berry's Brook (from the Lafayette Road crossing to the Brackett Road crossing) has not been undertaken by the CLG or NHDES.

EPA has established surface water screening levels (SLs) for PFOA and PFOS as guidance for assessing whether an unacceptable exposure risk is present for adults or children wading or playing in the Brook. With the exception of two surface water sampling locations near the perimeter of the landfill, concentrations of PFOA and PFAS are well below the SLs in all samples analyzed downstream of the Site.

It is unknown if the stocked brown trout and/or resident warm water fish species accumulate PFAS in fish tissue as a result of exposure to PFAS in Berry's Brook. The EPA has requested that CLG sample fish from Berry's Brook, downstream of the Site and analyze the fish tissue for the presence of PFAS to determine if fish consumption advisories are warranted.

2.1 Study Objectives

The overall goal of the fish tissue sampling and analysis is to collect composite samples of stocked brown trout, and two species of warm water fish (native to the brook), of a size that would be taken and eaten by recreational fishermen (i.e., 10 inches/254 millimeters (mm)), and analyze the composite samples for the presence of perfluorooctanoic acid (PFOA), Perfluorooctane sulfonate (PFOS), and perfluorobutane sulfonate (PFBS) in the fish tissue. (Note: the EPA has acknowledged that fish of the specified the target size may be difficult to capture and the actual size of collected fish may be adjusted based on actual sizes of fish collected during this effort).

To determine if background PFAS concentrations are present in brown trout, a composite sample (control sample) of five brown trout from the source hatchery (the Powder Mill Fish Hatchery in New Durham, New Hampshire), have been collected, processed and analyzed for PFAS. The hatchery fish samples were collected on April 16, 2018.

The objective of the field sampling effort will be to collect five similarly sized brown trout from Berry's Brook (Brackett Road and estuary) to compare PFAS concentrations in Brown Trout with EPA guidance.

Similarly, one composite sample of five similarly sized individual fish of each of two warm water species which may be expected to be harvested by recreational fishermen from the four stream (road crossing) locations would be prepared (a total of eight composite samples).

Specific handling, management, and analysis of composite samples and quality control samples (e.g., blanks, matrix spikes, duplicates, rinsates, etc.) are described in the attached Site-Specific Quality Assurance Project Plan (QAPP) (**Appendix A**).

2.2 Sampling Locations

Fish sampling for native warm water species will be undertaken at the four identified road crossing locations associated with Berry's Brook. The locations include:

- Lafayette Road (Route 1)
- Lang Road
- Sagamore Road
- Brackett Road

Sampling should begin at the crossing nearest the Site (Lafayette Road) and continue downstream.

Brown trout sampling will be undertaken at two locations including:

- Brackett Road
- Berry's Brook Estuary.

2.3 Sampling Schedule

Two separate sampling events will be completed as part of this sampling effort. The first sampling event will take place in late Spring. This event is being targeted to begin by June 15, 2018.

The second sampling event shall be targeted to brown trout only will be completed prior to the Fall stocking event, which is typically in mid-September. This later target date for brown trout will allow for an extended exposure time frame for fish stocked in April of 2018 as well as maintain an opportunity to catch returning trout from previous years if present.

The CLG shall initiate sampling for warm water, native species as early as practicable, but not later than June 15, and continue sampling until enough tissue has been collected and processed to perform adequate analysis. Specific dates will be determined based on brook flow conditions (i.e., during a period when near normal flow conditions exist and precipitation events are not anticipated that could significantly impact normal flow. The CLG shall initiate sampling for brown trout in the estuary portion of Berry's Brook (i.e., Brackett Road crossing) concurrent with warm water species sampling and again in mid-September, prior to the fall stocking event, and continue sampling until enough fish tissue (at least five fish) has been collected and processed.

The EPA has requested that the sampling for both events shall continue until sufficient samples of targeted species are collected or it is determined that collection of a sufficient number of suitable fish are unlikely to be obtained.

However, the CLG anticipates that the total field sampling efforts for both events will be completed over a two-three consecutive day period. If the requisite numbers of fish required as part of the sampling effort cannot be caught after the two to three-day field effort, field sampling will be curtailed and CLG will consult with the participating agencies to review the sampling results and determine a future course of action. Other considerations limiting the length of the field effort include, safe holding times to reduce unintended mortality of collected samples.

We plan to conduct up to one full day of sample collection at Lafayette, Lang and Sagamore Roads. Stream size is limited in each of these areas and a thorough fishing effort will be completed in this time frame. (Note: the crossing on Lafayette Road (Route 1) experiences high traffic volume with limited Brook access or vehicle pull-over space. For personnel safety, fishing is likely to be conducted during lower traffic volume periods (mid-morning to mid-afternoon and perhaps evenings (post rush hour)). If insufficient samples are collected during this one-day effort, CES will contact EPA and DES to review the efforts and determine if, and what type of additional effort is warranted at these locations.

Sampling efforts will be completed by a team of experienced personnel familiar with multiple methods of obtaining the targeted species.

Changes in project schedules and duration of sampling will be coordinated with the participating agencies.

3.0 | SAMPLING DESIGN

Berry's Brook flows in roughly northeasterly direction from the vicinity of the Site towards the Atlantic Ocean. The brook crosses several roads including Lafayette Road (closest to the Site), Lang Road, Sagamore Road, and finally Brackett Road where it begins forming an estuary and mixes with salt water from the Atlantic Ocean. It is assumed that these road crossings provide stream access for recreational fishing. As such, these four crossings have been identified by the EPA as sampling points for this effort. Each crossing provides unique physical features (e.g., channel definition, size and depth; bank type; and physical access) and accessibility which will determine the sampling methods. The CLG sampling team will evaluate each crossing and determine appropriate sampling methodology, and access. Sampling considerations include establishment of safe, nearby staging areas, where holding tanks equipped with aerators can be setup and maintained.

During each sampling event, researchers will use active or passive fish sampling gears to collect target fish species. Sampling for native, warmwater species in June will be conducted with a Smith Root LR20 (or similar) backpack electrofisher or Smith Root GPP 5.0 barge electrofisher. Sampling will take place at or near four road crossing locations in the study area, assuming they are safely accessible:

- Lafayette Road (Route 1)
- Lang Road
- Sagamore Road
- Brackett Road

As possible, sampling will begin at the crossing near Lafayette Road and continue downstream. Where feasible, block nets will be established at the bottom and top of 500-foot-long reaches to prevent escape of fish from the study areas.

Brown trout sampling in the spring and fall will be conducted using rod and reel, trolling, or gill netting at the following two locations:

- Brackett Road
- Berry Brook Estuary

Gill nets will be approximately 100 to 150-feet-long, 4 or 6 feet deep with 2.0 to 3.0-inch square mesh. Nets will be deployed perpendicular to water flow in the late afternoon and checked in the morning. Each net will have a weighted core-line so that they deploy properly. Additional weights will be added should tidal currents, wind, or river flow affect their orientation. Each gill net will be marked with fluorescent buoys to minimize the likelihood for recreational boats to become entangled. New Hampshire Fish and Game will be consulted with prior to initiating any gill netting regarding specific recommendations on gill net mesh size, time, location, and duration of deployment.

Captured individuals will be measured to the nearest millimeter and weighed to the nearest gram. Captured fish will be kept in live wells until they can be transferred to the interim processing laboratory for sample preparation. Smaller individuals will be collected in case individuals from the target size class (i.e., 10-inches+) are not captured. By-catch biological data (i.e., non-target species) will not be collected. Collected fish will be held and maintained alive, in aerated containers for the duration of each sampling day. At the end of each day, a determination will be made, with consultation with NH F&G and DES representatives, as to which fish will be kept as samples, and which will be returned to the collection site. In a effort to increase survival, fish will not be kept beyond the end of each sampling period.

The CLG will consult with NH F&G, EPA and DES representatives should field conditions warrant changes in collection methods, should initial efforts be unsuccessful.

3.1 Sampling Targets

The primary objective of the sampling effort is to investigate the occurrence of PFAS in edible tissue from harvestable sized adult fresh water fish that typically consumed by humans. The field objective is for the sampling teams to obtain one representative brown trout composite sample from the Brackett Road/estuary location, and one representative composite sample from two different warm water species from each sampling location.

Each composite sample shall consist of five, similar sized adult fish of the same species of a size to provide a minimum of 485 grams of edible tissue for analysis. Fish selected for each composite sample shall meet the following criteria:

- All are of the same species
- All satisfy legal requirements of harvestable size for the sampled stream, or at least be of consumable size (i.e., 10 inches/254 millimeters (mm)), if no legal harvest requirements are in effect (Note: Current New Hampshire fishing regulations indicate no length limits have been established for the species that could be expected to be part of this sampling effort)
- All are of similar size, so that the smallest individual in a composite is no less than 75 percent (%) of the total length of the largest individual
- All are collected at the same time (i.e., collected as close to the same time as possible, but no more than a week apart)

Individuals from different species will not be used in a composite sample.

The CLG shall retain fish smaller than 10-inches in the event that larger fish are not caught. These fish shall be frozen and stored for future laboratory analysis should an insufficient number of large fish be collected.

3.2 Sample Handling

Samples shall be collected and handled in accordance with the QAPP contained as **Appendix A**. Samplers shall ensure that PFAS-free equipment and supplies are used during the sampling events.

All tools used for processing samples shall be thoroughly cleaned and verified to be PFAS free.

Sampling teams shall record the following for each fish sample:

- Sample number
- Species name
- Specimen length
- Sampling location
- Sampling date

Each sample shall be individually wrapped in previously prepared aluminum foil (dull side in towards the fish). Place the sample in a food-grade polyethylene tubing or Ziploc bag and the bag labelled. All wrapped fish in the sample set from each location shall then be placed in a larger, sealed bag which shall be placed on wet ice for transport to the interim processing laboratory. At the end of each sampling day, collected samples will be transported to the interim processing laboratory. Samples shall be processed and handled for storage in accordance with the QAPP. Failure to process and maintain stored samples at appropriate temperatures will result in samples that cannot be analyzed. Appropriate Chain of Custody (CoC) documentation shall be followed with all samples.

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN (QAPP)

SENSIBLE SOLUTIONS

CES INC

DRAFT QUALITY ASSURANCE PROJECT PLAN (QAPP)

FOR

SAMPLE PREPARATION AND ANALYSIS

OF

PER-AND-POLYFLUOROALKYL SUBSTANCES (PFAS) IN FILLETS OF FISH FROM BERRYS BROOK NEAR COAKLEY LANDFILL SUPERFUND SITE

(Adapted by CES, Inc. from EPA Region 1, 2018)

FOR

COAKLEY LANDFILL GROUP

1 Junkins Avenue
Portsmouth, NH 03801

MAY 15, 2018
JN: 10424.018

Report Prepared By:
CES, Inc.
640 Main Street
Lewiston, Maine 04240

Corporate Office
465 South Main Street
PO Box 639
Brewer, Maine 04412
207.989.4824

www.ces-maine.com

Title and Approval Page

USEPA Region I QAPP Worksheet #1

Site Name/Project Name: Coakley Landfill Superfund Site

Site Location: North Hampton and Greenland, New Hampshire

Revision Number: 1

Revision Date: May 2018

Document Title: Quality Assurance Project Plan

Lead Organization (Agency, State, Tribe, Federal Facility, PRP, or Grantee): USEPA

Preparer's Name and Organizational Affiliation: CES, Inc., Consultant

Preparer's Address and Telephone Number: 640 Main Street, Lewiston, Maine 04240. (207) 795-6009

Preparation Date (Day/Month/Year): May 2018

Investigative Organization's Project Manager:

Peter Britz, City of Portsmouth/The Coakley Landfill Group, Coordinator Signature/Date

Sampling and Reporting Contractor / Quality Assurance Officer:

Michael Deyling, P.G., CES, Inc. Signature/Date

USEPA-Region I Remedial Project Manager

Richard Hull Signature/Date

USEPA Region I

NHDES Project Manager

Andrew Hoffman, PE Signature/Date

NHDES

TABLE OF CONTENTS

List of Acronyms and Abbreviations

C°	Celsius
CLG	Coakley Landfill Group
COC	Chain of Custody
DQO	Data Quality Objective
EAI	Eastern Analytical Laboratory
EPA	Environmental Protection Agency (also known as USEPA) ID Identification
GMP	Groundwater Management Permit
IR	Infra-red
MDL	Method Detection Limit
mL	milliliter
ML	Minimum Level
m/z	Mass-to-charge ratio of a specific ion monitored during high resolution mass spectrometric analyses, where m is the mass and z is the charge
NHDES	New Hampshire Department of Environmental Services
ng/g	nanograms per gram
ng/L	nanograms per liter
PFAS	Per- and Polyfluoroalkyl Substances
PFBS	Perfluorobutanesulfonic acid
PFHpA	Perfluoroheptanoic acid
PFHxS	Perfluorohexanesulfonic acid
PFNA	Perfluorononanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality control
QSA	Quality system audit
RfD	Reference Dose
RPD	Relative Percent Difference
SOP	Standard operating procedure
SOW	Statement of work
SPE	Solid-phase extraction
TBD	To Be Determined

QUALITY ASSURANCE PROJECT PLAN
COAKLEY LANDFILL SUPERFUND SITE
NORTH HAMPTON AND
GREENLAND, NEW HAMPSHIRE

SECTION 1 | INTRODUCTION

This Quality Assurance Project Plan (QAPP) defines Quality Assurance/Quality Control (QA/QC) procedures to be performed in support of fish tissue sampling activities at the Coakley Landfill Superfund Site (Site) located in North Hampton and Greenland, New Hampshire (see **Figure 3.1**) and updates the version completed in January 2018 by the US EPA Region 1.

The Coakley Landfill Group (CLG) identifies the laboratory selected for sample preparation as Eastern Analytical, Inc. (EAI) of Concord, New Hampshire and the laboratory selected for the analysis of per- and polyfluorinated alkyl substances (PFAS) in fish tissue as Vista Analytical Laboratory (Vista) of El Dorado Hills, California. This draft QAPP is based primarily on the January 2018 EPA draft version as well as memos dated February 22, 2018 and April 13, 2018 from Mr. Richard Hull, the Remedial Project Manager for the New Hampshire and Rhode Island Superfund Program with the US EPA, to Mr. Peter Britz, the Group Project Coordinator with the CLG.

This QAPP presents performance criteria, acceptance criteria, and objectives for the preparation and analysis of fish fillet tissue composite samples for the fish fillet tissue samples prepared under the QAPP that will be analyzed for PFAS. This QAPP also describes the methods and procedures that will be followed to ensure that the criteria and objectives are met. The scope of the initial QAPP was limited to fish sample preparation.

This QAPP was prepared in accordance with the most recent version of EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans* (USEPA 2001a), that was reissued in 2006. In accordance with EPA QA/R-5, this QAPP is a dynamic document that is subject to change as analytical activities progress. Changes to procedures in this QAPP must be reviewed by EPA Region 1 to determine whether the changes will impact the technical and quality objectives of the project. If so, the QAPP will be revised accordingly, circulated for approval, and forwarded to all project participants.

This QAPP is intended to be a project specific document applicable to fish tissue sampling activities at the Site. This plan, in conjunction with the Fish Tissue Sampling Field Work Plan, forms the framework upon which all fish tissue sampling activities will be conducted. The QAPP includes analytical methods and QA/QC review procedures that are considered to be applicable to these activities.

SECTION 2 | DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET

Table 2-1 presents a list of the individuals and organizations to whom the QAPP will be distributed. Each formal revision of this document will be dated and assigned a revision number to track the distribution of original and revised documents. A copy of this table will be maintained by the Sampling and Reporting Contractor throughout the duration of fish tissue sampling.

Table 2-2 provides the Project Personnel Sign-off Sheet. The Project Personnel Sign-off Sheet will be used to document that the project team (including off-site laboratory and data validation personnel) have read the QAPP and understand the requirements presented herein. The list will be revised as necessary as personnel are added to or leave the project team. Signed originals will be maintained by the Sampling and Reporting Contractor.

TABLE 2-1
QAPP DISTRIBUTION LIST
COAKLEY LANDFILL SUPERFUND SITE, NORTH HAMPTON AND GREENLAND, NH

QAPP Recipients	Title	Organization	Telephone Number
Richard Hull	USEPA Remedial Project Manager	USEPA Region I	617-918-1882
Andrew Hoffman	NHDES Project Manager	NHDES	603-271-6778
Peter Britz	Group Project Coordinator	Coakley Landfill Group	603-610-7215
Michael Deyling	Sampling and Reporting Contractor	CES, Inc.	207-795-6009
Michael Deyling	Quality Assurance Officer	CES, Inc.	207-795-6009
Jennifer Laramie	Laboratory Project Manager	Eastern Analytical, Inc.	603-410-3881

TABLE 2-2
PROJECT PERSONNEL SIGN-OFF SHEET
COAKLEY LANDFILL SUPERFUND SITE,
NORTH HAMPTON AND GREENLAND, NH

Project Personnel	Title	Organization	Signature	Date QAPP Read	QAPP Acceptable as Written?
Peter Britz	Group Project Coordinator	City of Portsmouth			
Suzanne Yerina	Field Operations Manager	CES, Inc.			
Michael Deyling	Sampling and Reporting Manager	CES, Inc.			
Michael Deyling	Quality Assurance Officer	CES, Inc.			
Jennifer Laramie	Laboratory Project Manager	Eastern Analytical			
Jennifer Miller	Laboratory Project Manager	Vista Analytical Laboratory			

SECTION 3.0 | PROJECT ORGANIZATION

The project management organization for fish tissue sampling at the Site provides a clear line of authority and a management control structure to support the program. This control structure provides:

- Clearly identified lines of communication;
- Management of two technical resources;
- Implementation of health and safety requirements; and
- Overall project quality assurance and quality control.

3.1 Project Organizational Chart

Figure 3-1 presents the organization structure of the project team for the fish tissue sampling program. The Group Project Coordinator will manage the activities of the Sampling and Reporting Contractor. The Sampling and Reporting Contractor will manage the laboratory for the fish tissue sampling program.

3.2 Communication Pathways

All formal communication between the Coakley Landfill Group (CLG); and USEPA and NHDES (the regulators) will be made through the CLGs' representatives, unless specific authorization is given to do otherwise. Field activities and project documentation will be scheduled and implemented by the Sampling and Reporting Contractor. The Sampling and Reporting Contractor will report directly to the CLG and will review technical information and prepare documents for submittal to USEPA and NHDES.

3.2.1 Modifications to Approved QAPP

Modifications to this QAPP may occur. Modification to the approved QAPP will be made whenever a project activity requires significant modification to achieve project goals. Activities that will require a modification or addendum submittal may include:

- Changes to sample collection procedures or locations;
- Changes to sample collection and analysis procedures;
- Change in sampling or reporting contractor and/or change in analytical laboratory used for analysis; and
- Change in key project personnel.

Changes will only be implemented after review and approval by USEPA and NHDES. Verbal approval may be necessary to expedite project execution. Verbal approvals will be documented and submitted for formal approval as soon as possible. When formal revisions of this document are made, the revision number will be updated, and the Group Project Coordinator will update the QAPP Distribution List.

3.3 Personnel Responsibilities and Qualifications

Key personnel during implementation of the fish tissue sampling program (shown on **Table 3-1**) include: USEPA Remedial Project Manager, NHDES Project Manager, NHDES Quality Assurance Coordinator, Group Project Coordinator, Sampling and reporting Manager, Quality Assurance Officer, Sampling Staff, Field Coordinator/Field Quality Assurance Officer, and Analytical Project Managers.

USEPA Remedial Project Manager

The Remedial Project Manager for the Site is shown in **Table 3-1**. The primary responsibilities of the Remedial Project Manager include administration of USEPA responsibilities and oversight of the activities conducted under the Consent Decree.

NHDES Project Manager

The NHDES Project Manager for the Site is shown in **Table 3-1**. The primary responsibilities of the NHDES Project Manager include administration of NHDES responsibilities and oversight of the activities conducted under the Consent Decree and the Groundwater Management Permit (GMP).

Coakley Landfill Group

The Coakley Landfill Group (CLG) maintains ultimate responsibility for project completion. The Group Project Coordinator serves as the main point of contact between the USEPA/NHDES and the Sampling and Reporting Contractor.

Sampling and Reporting Contractor

The Sampling and Reporting Contractor will be responsible for performing fish tissue sampling under this QAPP and the Field Work Plan. Members of the Sampling and Reporting Contactor team include:

- Sampling and Reporting Manager
  The Sampling and Reporting Manager will determine the technical staff involved with technical review, project management, and QA/QC activities.
- Quality Assurance Officer
  The Quality Assurance Officer's (QAO's) responsibilities will include overseeing the analytical chemistry program and assessing the general usability of the data generated.
- Project Staff
  Project Staff will be selected by the sampling and reporting manager to perform the tasks described in this QAPP and the Field Work Plan.

Analytical Laboratories

The Analytical Laboratories will be responsible for providing preparation of fish samples and analytical services and ensuring that quality assurance and quality control practice are maintained for analytical work.

3.4 Special Training Requirements/Certification

Training of field personnel will be provided by the Sampling and Reporting Contractor. Routine training will be completed at the beginning of each field event. The Sampling and Reporting Contractor will review all applicable procedures with the field personnel to verify that the project requirements and procedures are understood.

SECTION 4.0 | PROJECT PLANNING/PROBLEM DEFINITION

This QAPP provides guidance and specifications to ensure that project planning is performed in a consistent manner from task to task. The QAPP has been developed to ensure the following:

- The samples are maintained under controlled conditions using appropriate and documented procedures;
- Samples are uniquely identified and controlled through sample tracking systems and chain-of-custody (COC) protocols;
- Laboratory and analytical results are of known quality, consistent with Data Quality Objectives (DQOs), and compatible with USEPA analytical procedures through the use of appropriate analytical methods, preventative maintenance, calibration and analytical protocols, quality control (QC) measurements, review, and correction of analytical problems;
- Calculations and evaluations are accurate, appropriate, and consistent throughout the project; and,
- Records are retained as documentary evidence of the quality of samples, applied processes, equipment, and results.

4.1 Problem Definition/Site History and Background

The Coakley Landfill Site description and background is provided in **Section 2.0** (Project Background and Description) of the Field Work Plan.

SECTION 5.0 | PROJECT OVERVIEW AND SCHEDULE

5.1 Project Overview

The purpose of the fish tissue sampling program is to investigate the occurrence of PFAS in the edible tissues (fillets) of harvestable-sized adult freshwater fish, and possibly saltwater fish, that are typically consumed by humans. The overall goal of the fish tissue sampling and analysis is to collect composite samples of stocked brown trout and two species of warm water fish (native to Berrys Brook), of a size that would be taken and eaten by recreational fishermen (i.e., 10 inches (in)/254 millimeters (mm)), and analyze the composite samples for the presence of the six PFAS compounds that area currently analyzed by the CLG for surface water, including perfluorobutanesulfonic acid (PFBS), perfluoroheptanoic acid (PFHpA), perfluorohexanesulfonic acid (PFHxS), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), and perfluorooctanesulfonic acid (PFOS). The results for PFOA, PFOS, and PFBS will be compared against site-specific risk-based screening levels (SLs) to evaluate whether further risk evaluation is appropriate.

5.1.1 Sampling Tasks

Fish tissue sampling may include the following tasks:

- Collecting one composite fish sample of each of two warm water species which may be expected to be harvested by recreational fishermen from four stream (road crossing) locations as outlined in Section 2.2 in the Field Work Plan (a total of eight composite samples);
- Collecting two composite samples of brown trout (one from the hatchery and one from locations listed in Section 2.2 of the Field Work Plan);
- Shipping whole fish samples to an interim frozen storage facility (EAI);
- Transferring the whole fish samples to a laboratory for fish sample preparation, which includes collecting and freezing the head for potential aging of scales, filleting the fish, homogenizing the fillet tissue composites, and preparing fillet tissue aliquots for analysis of specific PFAS identified in this QAPP; and
- Analyzing the fillet tissue samples for PFAS.

Ideally, each composite fish sample is a routine fish composite sample that consists of five fish of adequate size to provide a minimum of 485 grams of edible tissue for analysis. Fish will be selected for each composite sample by applying the criteria outlined in Section 3.1 of the Field Work Plan.

SECTION 6.0 | PROJECT QUALITY OBJECTIVES AND PERFORMANCE CRITERIA

The overall quality objective for the analysis of fish fillet tissue samples for PFAS is to produce data of known and documented quality. Completeness is defined as the percentage of samples collected in the study for which usable analytical results were produced. The goal for completeness is 95% and it is calculated at the sample-analyte level, such that an issue with the quality of one analyte out of many does not invalidate the entire sample.

The methods and quality control acceptance criteria employed by the laboratories under contract for analyses of fish fillet tissue samples for PFAS are described in **Sections 8.4 and 8.5** of this QAPP. Data usability for each analysis will be assessed using processes described in **Section 9** and the QC criteria summarized in **Section 8.5** of this QAPP.

Fish Tissue Sample Preparation

All laboratory staff involved in the preparation of fish tissue samples must be proficient in the associated tasks, as required by the Coakley Fish Procedure dated May 10, 2018, prepared by Vista (**Appendix A**).

Specialized training will be provided for laboratory technicians who will be preparing fish tissue fillets and homogenates for this project. This training will be conducted at the sample preparation laboratory for all laboratory staff involved with fish tissue sample preparation to accomplish the following objectives:

Analysis of Fish Tissue Samples

All laboratory staff involved in the analysis of fish tissue samples must be proficient in the associated tasks, as required by each analytical laboratory's existing quality system. All contractor staff involved in analytical data review and assessment will be proficient in data review, and no specialized training is required for data reviewers for this project.

SECTION 7.0 | DOCUMENTS AND RECORDS

The Statements of Work (SOWs) for the analytical subcontracts will provide the specific requirements for laboratory deliverables. The major points are summarized below:

- The laboratory must provide reports of all results required from analyses of environmental and QC samples.
- Summary level data must be submitted in electronic format and must include the following information: sample number, analyte name and CAS number, laboratory sample ID, measured amount, reporting limits and units, sample preparation date, and analytical batch ID (if applicable).
- The laboratory shall provide raw data in the form of direct instrument readouts with each data package. Raw data include:

    - Copy of traffic report, chain-of-custody records, or other shipping information
    - Instrument readouts and quantitation reports for analysis of each sample, blank, standard and QC sample, and all manual worksheets pertaining to sample or QC data or the calculations thereof
    - Copies of bench notes, including preparation of standards and instrumental analyses

The laboratories will maintain records and documentation associated with these analyses for a minimum of five years after completion of the study.

SECTION 8.0 | DATA GENERATION AND ACQUISITION

8.1 Sampling Process Design (Experimental Design)

The primary objective of the program is to investigate the occurrence of, PFAS in the edible tissue (fillets) of harvestable-sized adult freshwater fish, and possibly saltwater fish that are typically consumed by humans. The study will provide:

- Statistically representative data on the concentrations of PFAS in fish commonly consumed by humans

To meet the study objectives, one composite fish sample is collected from each site. Ideally, each fish sample is a routine fish composite sample that consists of five fish of adequate size to provide a minimum of 485 grams of edible tissue for analysis. Fish are selected for each composite sample by applying the following criteria:

- All are of the same species
- All satisfy legal requirements of harvestable size (or weight) for the sampled stream, or at least be of consumable size if no legal harvest requirements are in effect
- All are of similar size, so that the smallest individual in a composite is no less than 75% of the total length of the largest individual
- All are collected at the same time, i.e., collected as close to the same time as possible, but no more than one week apart (Note: Individual fish may have to be frozen until all fish to be included in the composite are available for delivery to the designated laboratory)

Accurate taxonomic identification is essential in preventing the mixing of closely related target species. Under no circumstances are individuals from different species to be used in a composite sample.

8.2 Sampling Methods

Sampling method procedures and requirements for collection of human health fish samples are summarized below.

The field objective is for sampling teams to obtain one representative fish composite sample from each sampling site. Collecting fish composite samples is a cost-effective means of estimating average chemical concentrations in the tissue of target species, and compositing fish ensures adequate sample mass for analysis of chemicals. The sampling procedures specify that each composite should consist of five similarly sized adult fish of the same species. The method applied for fish collection is left to the discretion of the field team, but it typically involves angling, gillnetting, electrofishing, and occasionally trawling.

PFAS-free gloves should be used during handling and processing of fish. Sampling personnel should avoid the use of potentially PFAS containing food wrappers, water resistant notebooks, Tyvek, Teflon, and potentially PFAS containing clothing (such as raingear) unless it has been washed several times. Prior to usage, batches of all disposable materials used for handling, storing, sealing, processing, and labeling samples should be verified to be PFAS free by rinsate sampling and analysis. Tools for processing fish should be thoroughly cleaned and verified to be PFAS free by rinsate sampling and analysis.

In preparing fish samples for shipping, field teams record sample number, species name, specimen length, sampling location and sampling date on a fish collection form. Each fish is wrapped in solvent-rinsed aluminum foil, with the dull side in using foil sheets. Individual are still frozen and in good condition;

- Check the temperature of one of the samples in the cooler using a thermometer that reads to -20 degrees Celsius (ºC) or less, or an infra-red (IR) temperature "gun" and records the reading on the sample tracking form;
- Verify that all associated paperwork is complete, legible, and accurate;
- Compare the information on the label on each individual fish specimen to the sample tracking form for each composite and verifies that each specimen was included in the shipment and is properly wrapped and labeled;
- Notify Vista of the fact that samples were received and of any discrepancies in the paperwork identified above; and,
- Transfer the samples to the freezer for long-term storage

The sample preparation laboratory notifies EAI immediately about any problems encountered upon receipt of samples. Problems involving sample integrity, conformity, or inconsistencies for whole fish samples are required to be reported to EAI in writing (e.g., by email) as soon as possible following sample receipt and inspection.

The sample preparation laboratory must store the fish sample frozen to less than or equal -20 °C until they are distributed to the laboratory performing tissue preparation and analyses.

Shipment of Whole Fish Samples to the Analytical Labs

EAI will be responsible for oversight of shipping the whole fish samples from the sample preparation laboratory to the analytical laboratory. The whole fish samples will be packaged in sturdy coolers for shipping and wrapped with bubble wrap or other suitable packaging to protect the samples in transit. Whole fish samples will be shipped frozen with sufficient dry ice in the coolers to ensure that the samples remain frozen for at least 48 hours. EAI will prepare sample tracking paperwork and include it in each shipment.

When received at the respective analytical laboratories, the whole fish samples are inspected for damage, logged into the laboratory, and immediately placed into freezers. Because the samples are shipped frozen, typical temperature blanks consisting of a bottle of water are not practical (they may break due to expansion), so they are not required. The laboratories measure and record the temperature of the coolers containing the samples on receipt using an infrared temperature sensor or other suitable device. EAI is notified of the receipt of the whole fish samples by email. Vista will advise EAI of whole fish sample receipt. Any questions from the laboratories regarding sample paperwork or condition will be sent to EAI, routed to Vista as appropriate, and EAI will send the answers back to the appropriate laboratory.

Whole fish samples will be stored frozen at less than or equal to -20^{o} C until prepared and analyzed. There are no formal holding time studies or requirements that apply to PFAS. For PFAS, Vista will use a one-year holding time for the fish tissue samples, because there is no evidence that indicates that there are practical limitations regarding the loss of PFAS from tissues in that time frame. EPA will note any results for PFAS generated outside of these 1-year holding times, but this will not preclude use of these results for the purposes of this project.

8.3 Methods

8.3.1 Fish Sample and Rinsate Preparation

Fish Sample Preparation

Vista is responsible for filleting each valid fish sample, homogenizing the fillet tissue, and preparing the required number of fillet tissue aliquots for analysis. The specific procedures for fish sample preparation activities are described in **Appendix A**.

Fish are filleted by qualified technicians using thoroughly clean utensils and cutting boards (cleaning procedures are detailed in **Appendix A**). Each fish is weighed to the nearest gram wet weight, scaled, rinsed with deionized water, and filleted on a glass cutting board. Fillets from both sides of each fish are prepared with scales removed, skin on, and belly flap (ventral muscle and skin) attached. Fillets are composited using the "batch" method, in which fillets from all of the individual specimens that comprise the sample are homogenized together, regardless of each individual specimen's proportion to one another (as opposed to the "individual" method, in which equal weights of fillets from each specimen are added together). The latter method was not used because it was not described in the EPA QAPP and it is uncertain whether there will be enough fish of edible size to provide the required tissue mass.

An electric meat grinder is used to prepare homogenate samples. Entire fillets (with scale-free skin and belly flap) from both sides of each fish are homogenized for an approximately equal and recorded period of time, and the entire homogenized volume of all fillets from the fish sample is used to prepare the tissue sample. Tissues are mixed thoroughly until they are completely homogenized as evidenced by a fillet homogenate that consists of a fine paste of uniform color and texture. In the EPA 2018 Draft QAPP, each sample, as well as some split samples were required to be analyzed for lipids by total fatty acid analysis. This was required to demonstrate that homogenization was comparable between samples, as well as to enable lipid normalization of lipophilic PCB and dioxin concentrations. Lipid analysis was considered to be unnecessary for this preliminary study given that the samples will be homogenized for an equal time to a uniform color and texture. The collective weight of the homogenized tissue from each sample is recorded to the nearest gram (wet weight) after processing. The sample preparation laboratory prepares fillet tissue aliquots according to the specifications listed in the fish sample preparation procedures in **Appendix A**.

Rinsate Preparation

As part of the fish sample preparation process, the sample preparation laboratory will create rinsates of the equipment used to homogenize fillet tissue samples. Two of these rinsates will be analyzed for PFAS. The results of the rinsates will be used to assess the ongoing effectiveness of the laboratory's equipment cleaning procedures.

8.3.2 PFAS Analysis of Fillet Tissue and Rinsate Samples

There are no formal analytical methods from EPA or any voluntary consensus standards bodies for the PFAS analyses of tissue samples. Therefore, fish tissue samples will be analyzed by Vista Analytical Laboratory (El Dorado Hills California), or other suitable laboratory, using procedures developed, tested, and documented in that laboratory. A copy of the Vista SOP will be made available to EPA for review on request. The analytical procedures are briefly described below, based on information in the SOP. The 6 target PFAS analytes are listed in **Appendix B**. Although there are other PFAS that may be detectable, this study is limited to the identified six target PFAS compounds that area currently analyzed by the CLG for surface water. This is adequate for any eventual risk evaluation because there are oral toxicity values (i.e. oral reference doses, or RfDs) only for PFOA, PFOS and PFBS.

The concentration of each PFAS is determined using the responses from one of the ^{13}C- or ^{18}O- labeled standards added prior to sample extraction, applying the technique known as isotope dilution. As a result, all of the target analyte concentrations are corrected for the recovery of the labeled standards, thus accounting for extraction efficiencies and losses during cleanup. (Because a labeled standard for perfluorobutanesulfonic acid is not commercially available, this target analyte is quantified using the response for ^{18}O-labeled perfluorohexanesulfonic acid, a closely related compound.)

Approximately 2 g of fish tissue are required for analysis. (If matrix-related analytical problems are identified during the analysis of a given fish tissue sample, a sample aliquot of 1 g may be used to minimize those problems.) The sample is spiked with eight isotopically labeled standards and extracted by shaking the tissue in a caustic solution of methanol, water, and potassium hydroxide. The hydroxide solution breaks down the tissue and allows the PFAS to be extracted into the methanol/water.

After extraction, the solution is centrifuged to remove the solids and the supernatant liquid is diluted with reagent water and processed by solid-phase extraction (SPE). The PFAS are eluted from the SPE cartridge and the eluent is spiked with additional labeled recovery standards and analyzed by high performance liquid chromatography with tandem mass spectrometry.

The aqueous rinsate samples will be analyzed using a procedure based on EPA Method 537 from the Office of Groundwater and Drinking Water (USEPA 2009). The 250-mL aqueous rinsate sample is spiked with the labeled standards and processed by SPE, in a similar manner as is used for the tissue samples. The PFCs are eluted from the SPE cartridge and the eluent is spiked with additional labeled recovery standards and analyzed by high performance liquid chromatography with tandem mass spectrometry.

Tissue sample results are reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g). Method detection limits and Minimum Levels for PFAS are listed in **Appendix B**. Aqueous rinsate results are reported based on the volume of the rinsate sample, in nanograms per liter (ng/L).

8.4 Fish Sample Preparation and Analytical Quality Control

The fish fillet tissue sample preparation procedures being applied are specific to the project but are based on the procedures specified by previous fish tissue studies. The associated quality control activities are described in **Subsection 8.5.1**.

The analytical procedures being applied by the laboratories designated for analysis of fillet tissue samples will include many of the traditional EPA analytical quality control activities. For example, all samples are analyzed in batches and each batch includes:

- Up to 20 field samples and the associated QC samples
- Blanks – 5% of the samples within a batch are method blanks

Other common quality control activities vary by the analysis type, and they will be described in the subsections below as the method-specific information becomes available. The QC activities associated with the PFAS analyses are described in **Subsection 8.5.2**.

8.4.1 Fish Sample Preparation

The project-specific QC procedures for fish sample preparation include preparation and testing of equipment rinsate samples. The rinsate samples are prepared and analyzed individually, not in batches of up to 20, in order to provide timely feedback of the cleanliness of the homogenization equipment. Therefore, the quality control samples associated with the rinsate samples analyzed for PFAS are usually analyzed with each rinsate sample.

8.4.2 PFAS Analysis Quality Control

Quality control samples associated with each batch of tissue samples or rinsate samples analyzed for PFAS are summarized in **Table 8-1** below.

Table 8-1
QC Samples and Acceptance Criteria for PFAS Analysis of Tissues and Rinsates

QC Operation	Frequency	Acceptance Limit	Corrective Action
Labeled Compounds	Spiked into every sample before extraction	Per Appendix B of this QAPP	Evaluate failure and impact on samples. If sample results are non-detects for analytes which have a high labeled compound recovery, report non-detect results with case narrative comment. For detected analytes with low labeled compound recovery, extract and analyze a smaller sample aliquot.
Calibration Verification	Every 12 hours, before sample analysis.	Per Appendix B of this QAPP	Evaluate failure and impact on samples. If sample results are non-detects for analytes which have a high bias, report non-detect results with case narrative comment. *or* Immediately analyze two additional consecutive verification standards. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable verification standard.
Lab Control Sample (LCS)	Once per batch of up to 20 field samples	Per Appendix B of this QAPP	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and sample results are non-detects, report with case narrative comment. If LCS has low bias, or if there are detected analytes with failures, evaluate and re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes.
Method blank	Once per batch of up to 20 field samples	Less than or equal to the MDLs in Appendix B of this QAPP	As noted elsewhere, all results, including blanks, are reported down to the method detection limit (MDL). ◆ If the method blank result for any PFC is above the MDL, but below the laboratory's nominal quantitation limit, the laboratory will flag all associated tissue sample and rinsate results as having a detectable method blank for that analyte. (Subsequent validation of the results by EPA or its contractors will evaluate the potential contribution of the blank to such sample results.) ◆ If the method blank result is above the quantitation limit, the laboratory will reanalyze the method blank. - If the method blank reanalysis result is below the quantitation limit, then the laboratory will reanalyze all of the associated tissue or rinsate samples and QC samples. - If the method blank reanalysis result is still above the quantitation limit, then the laboratory will re-extract and reanalyze all tissue or rinsate samples with original results above the MDL.
Laboratory duplicate	Once per batch of up to 20 field samples	The relative percent difference (RPD) of the duplicate measure-ments must be < 40%	Evaluate the data, and re-extract and reanalyze the original sample and duplicate: ◆ If the reanalysis results meet the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples. ◆ If the reanalysis result still does not meet the RPD limit, then the laboratory will re-extract and reanalyze all field samples with original results above the MDL.

8.5 Instrument/Equipment Testing, Inspection, and Maintenance

There are no analytical instruments used in the preparation of the fillet tissue samples. However, the balances used to weigh the whole fish, the fillets, the homogenized fillet tissue, and the fillet tissue aliquots are inspected and serviced on a regular schedule and the homogenization equipment (i.e., meat grinder) will be inspected when it is reassembled after cleaning between samples.

All analytical instrumentation associated with the rinsate analyses and fillet tissue sample analyses will be inspected and maintained as described in the respective analysis methods and laboratory SOPs.

8.6 Instrument/Equipment Calibration and Frequency

The balances used to weigh the whole fish and the fillet tissue during the various stages of homogenization and aliquot preparation will be calibrated on a regular schedule and calibrations are verified at the beginning of each day on which the balances are used.

All analytical instrumentation associated with the rinsate analyses and fillet tissue sample analyses will be calibrated as described in the respective analysis methods. The methods in **Table 8-1** for the rinsate analyses require multi-point initial calibrations and periodic calibration verifications, and all the methods contain QC acceptance criteria for calibration.

8.7 Inspection/Acceptance of Supplies and Consumables

The inspection and acceptance of any laboratory supplies and consumables associated with the rinsate analyses and fillet tissue sample analyses are addressed in the individual laboratory operating procedures to be used, and/or in the laboratory's existing overall quality system documentation. There are no additional requirements specific to this project, and therefore, none are described here.

8.8 Data Management

Data management practices employed in this study will be based on data management practices to be described.

The laboratory is accredited for PFAS analyses in tissue by the Department of Defense Environmental Laboratory Accreditation Program, the New Jersey Department of Environmental Services, and the Oregon Environmental Laboratory Accreditation Program.

8.9 Reports to Management

The sample preparation laboratory will provide status report that describes all of the fish samples processed during the previous week.

Following data verification and validation of all project analytical data, Vista will apply standardized data qualifier flags to the fish tissue results in the project database that describe data quality limitations and recommendations concerning data use.

SECTION 9.0 | DATA VALIDATIOIN AND USABILITY

9.1 Data Review, Verification, and Validation

The data review, verification, and validation aspects of the fillet tissue sample preparation effort are more limited than those that will be applied to the PFAS analysis efforts. However, the procedures described below apply to both types of data.

9.1.1 Data Review

All laboratory results and calculations will be reviewed by the Laboratory Manager prior to data submission. Any errors identified during this peer review will be returned to the analyst for correction prior to submission of the data package. Following correction of the errors, the Laboratory Manager will verify that the final package is complete and compliant with the contract and will sign each data submission to certify that the package was reviewed and determined to be in compliance with the terms and conditions of the contract.

9.1.2 Data Verification

The basic goal of data verification is to ensure that project participants know what data were produced, if they are complete, if they are contractually compliant, and the extent to which they meet the objectives of the study. Every laboratory data package submitted under this study will be subjected to data verification. The verification process is designed to identify and correct data deficiencies as early as possible in order to maximize the amount of usable data generated during this study.

References

USEPA. 2018. Draft Quality Assurance Project Plan (QAPP) for Sample Preparation and Analysis of Per-and Polyfluoroalkyl Substances (PFAS) in Fillets of Fish from Berrys Brook near Coakley Landfill Superfund Site. Prepared January 22, 2018.

Vista Analytical Laboratory. 2018. Coakley Fish Procedure. Revision 0.0. May 10, 2018.

TABLE 3-1

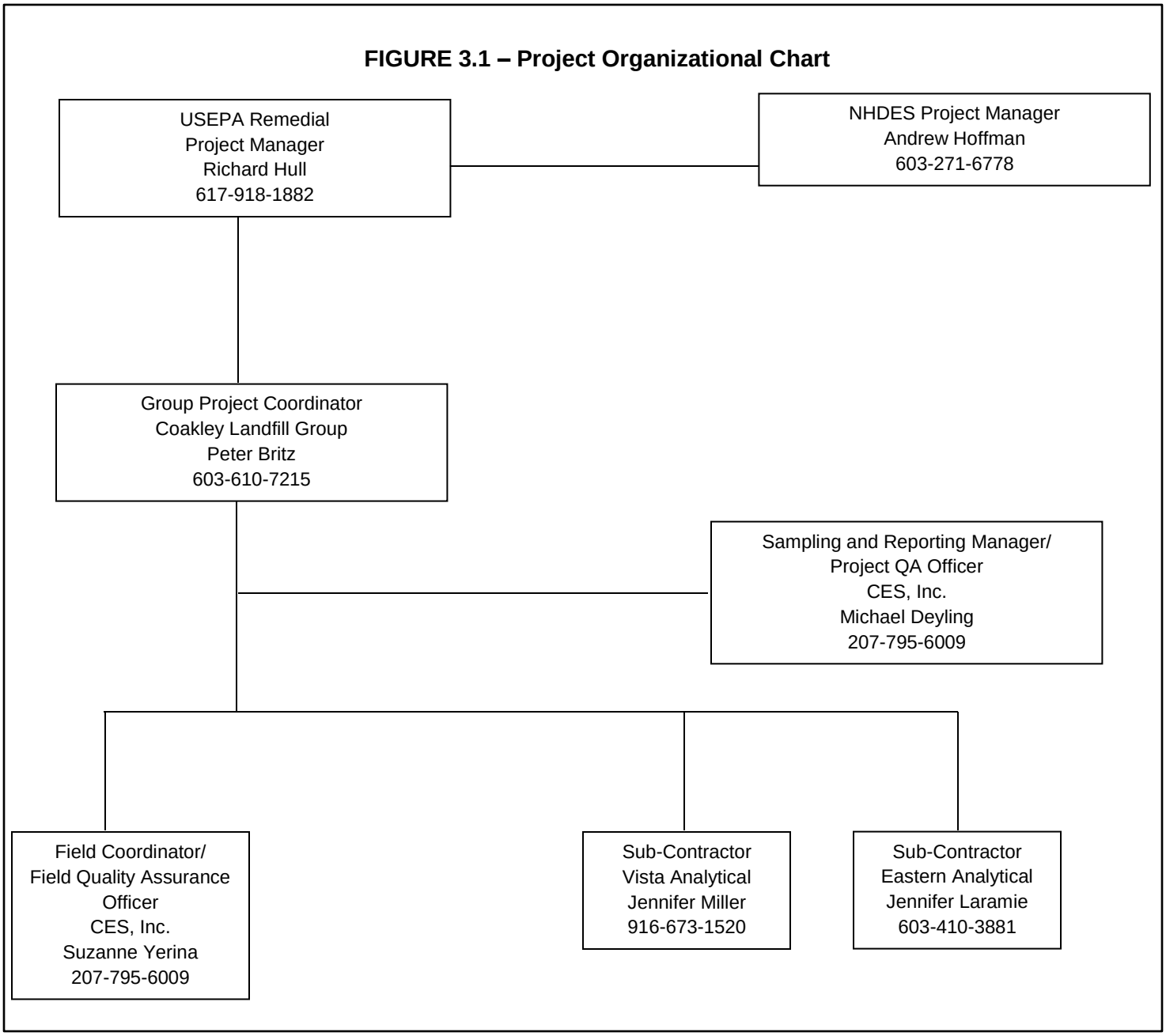
COAKLEY LANDFILL SUPERFUND SITE PROJECT ORGANIZATION

TABLE 3-1
COAKLEY LANDFILL SUPERFUND SITE
PROJECT ORGANIZATION

Individual	Title	Organization	Telephone Number
Richard Hull	USEPA Remedial Project Manager	USEPA New England (Region 1)	(617) 918-1882
Andrew Hoffman	NHDES Project Manager	NHDES	(603) 271-6778
Sharon Perkins	NHDES Quality Assurance Coordinator	NHDES	(603) 271-6805
Peter Britz	Project Coordinator	The Coakley Landfill Group	(603) 610-7215
Michael Deyling	Sampling and Reporting Manager/Quality Assurance Officer	CES, Inc.	(207) 795-6009
Dennis Kingman	Sampling Staff	CES, Inc.	(207) 944-2700
Suzanne Yerina	Field Operations Manager	CES, Inc.	(207) 841-1387
Jennifer Laramie	Analytical Laboratory Project Manager	Eastern Analytical, Inc.	(800) 287-0525
Jennifer Miller	Analytical Laboratory Project Manager	Vista Analytical Laboratory	(916)-673-1520

FIGURE 3-1

FIGURE 3.1 – Project Organizational Chart



APPENDIX A

COAKLEY FISH PROCEDURE

1. Introduction:

This document provides information on fish sample preparation for the Coakley Fish Project

2. Area Prep

Thoroughly clean utensils and cutting boards

- Wash with detergent solution and warm tap water in the sink
- Rinse three times with tap water
- Rinse three time with DI water
- Perform 4 solvent rinse followed by MeOH
- Final rinse three times rinse with DI water

NOTE: detergent must be phosphate and scent free

Cover all surfaces in aluminum foil

- Cover hood surface with new hood paper
- Place new aluminum foil over the hood paper
- Cover cutting boards with at least two layers of aluminum foil

NOTE: Repeat this procedure between scaling and filleting fish to avoid cross-contamination

3. Inspecting the fish sample

- Confirm that the specimen ID on fish and tracking form match (reconcile sample)
- Check fish for damage: damage includes cuts in flesh or leaking guts
    - Document any damage on bench sheet and notify PM to notify client

4. Prep Fish

- Thaw fish slightly in hood: flesh should still have ice crystals in the muscle tissue when prepping. Do not let sample thaw all the way.
- Take fish out of aluminum foil and weigh whole fish
- Document weight.
- Rinse whole fish with DI water to remove slime and any debris from the fish
    - Blot with Kim wipe to remove any stubborn debris
- Scale fish by scraping backwards up the fish starting from the tail using a clean knife
    - Remove as many scales as possible, they are difficult to homogenize

5. Fillet Fish

- Change all foil and clean all utensils using the procedure from section 2, Area Prep
- Ensure that all dried on scales are cleaned off of hood to avoid contamination
- Ideally, fish should be filleted when ice crystals are still present in the muscle tissue
- Fillet fish by making an incision from the head down the length of the spine, cutting parallel to the gills, and then slicing off the fillet including the belly flap
- Special care must be taken to NOT puncture any of the organs near the belly flap to prevent contamination of the fillet.

- If any organs are inadvertently punctured then immediately rinse the fish with DI water and record the incident on the log sheet
- Remove any bones from the fillet
- Repeat the process for all five fillets
- Weigh all five fillets together before moving on to homogenization
- Record total weight on the log sheet

6. Homogenize the samples

- Begin by scoring the skin of the fillet as small as possible, using cross hatching motions
- Cut the fillet into cubes to ensure easy grinding
- Homogenize fillet cubes in grinder (make sure grinder has been cleaned according to procedure in section 2, Area Prep)
- Record the amount of time and the speed that was used to homogenize sample in the log sheet
- Mix all five homogenates together thoroughly until paste of equal texture and color is formed.
  - **NOTE: no chunks of skin or tissue are acceptable**
  - Note: if skin bits do not fully homogenize, use the fish scissors to cut the skin bits into small chunks (usually around 2-3 mm)
  - Note: dry ice chunks may be used to help grind stubborn fish
- Record the net weight of all 5 homogenized fillets together (at least 485 g must be present to proceed with extraction)

ALIQUOTING SAMPLES

- Use a 50 ml HDPE jar
- Print labels and adhere them to the jars
  - **NOTE: ensure label includes the site ID number, sample ID number, analysis type, aliquot weight, prep batch ID, and prep date**.
- Seal label with clear tape
- Weigh the empty jar and record the value
- Weigh out 10-15 g of sample taken from the large homogenate mass which consists of all five fillets mixed together
  - ensure homogenate mass is adequately homogenized for a representative sample
- Weigh the jar with the sample in it and record that mass. Calculate the mass of the sample.
- Ensure jar outsides are clean and place inside of plastic freezer bag to be stored in freezer

APPENDIX B

QUALITY CONTROL ACCEPTANCE CRITERIA FOR PFAS ANALYSIS OF TISSUE AND RINSATE SAMPLES

Calibration Verification (VER), Laboratory Control Sample (LCS), and Labeled Compound Recovery QC					
		LCS Recovery (%)		Labeled Compound Recovery in Samples	
Analyte	VER (%)	Tissues	Rinsates	Tissue	Rinsate
PFHpA	70-130	70-130	80-120		
PFOA	70-130	70-130	80-120		
PFNA	70-130	70-130	80-120		
PFBS	70-130	70-130	70-130		
PFHxS	70-130	70-130	70-130		
PFOS	70-130	70-130	70-130		
Quantitation Standards					
$^{13}C_2$-PFOA	40-150	40-150	40-150	40-150	40-150
$^{13}C_5$-PFNA	40-150	40-150	40-150	40-150	40-150
$^{18}O_2$-PFHxS	40-150	40-150	40-150	40-150	40-150
$^{13}C_4$-PFOS	20-130	20-130	40-150	20-130	40-150
Cleanup Standard					
$^{13}C_8$-PFOA	40-150	40-150	40-150	40-150	NA

FIGURE V101

FISH SAMPLING LOCATIONS

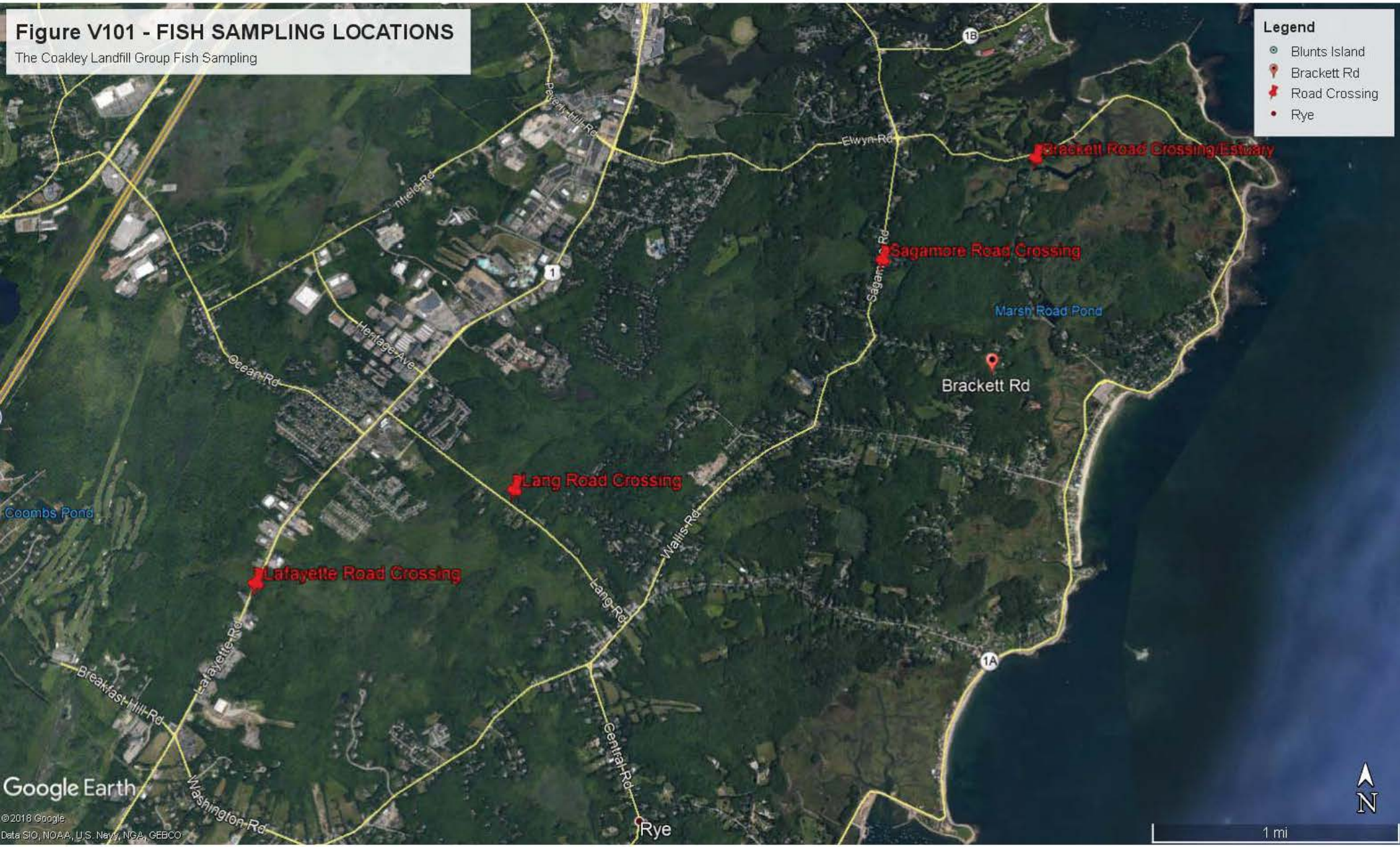


Figure V101 - FISH SAMPLING LOCATIONS
The Coakley Landfill Group Fish Sampling
Legend
Blunts Island
Brackett Rd
Road Crossing
Rye
Brackett Road Crossing/Estuary
Sagamore Road Crossing
Marsh Road Pond
Brackett Rd
Lang Road Crossing
Coombs Pond
Lafayette Road Crossing
Peverly Hill Rd
Elwyn Rd
Sagamore Rd
Heritage Ave
Ocean Rd
Wallis Rd
Lang Rd
Lafayette Rd
Breakfast Hill Rd
Central Rd
Washington Rd
Rye
1B
1
1A
Google Earth
©2018 Google
Data SIO, NOAA, U.S. Navy, NGA, GEBCO
N
1 mi