

Quality Assurance Project Plan

For the PFAS Drinking Water Sampling Program
Chemours Chambers Works
Deepwater, New Jersey

Submitted on behalf of:
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Submitted by:
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1.0 Introduction

This PFAS Drinking Water Sampling Program Quality Assurance Project Plan (QAPP) is written to guide sampling and analysis for perfluoroalkyl substances (PFASs) for drinking water samples that will be collected in the vicinity of the Chambers Works site boundary.

This QAPP is written to describe policies, project organization, functional activities, and quality assurance/quality control (QA/QC) measures intended to achieve the data quality objectives for sampling activities associated with the drinking water sampling project.

This QAPP is intended to meet the requirements for conducting the work in accordance with QA/QC field protocols for collecting environmental measurement data.

This QAPP was prepared in general accordance with the following U.S. Environmental Protection Agency (USEPA) documents:

- Uniform Federal Policy for Quality Assurance Project Plans, Optimized UFP-QAPP Worksheets, Intergovernmental Data Quality Task Force (March 2012)
- EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA/240/B-1/003, March 2001)
- EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5 (EPA/240/R-02/009, December 2002)

2.0 QAPP Organization

The QAPP consists of the table of contents, the introduction, the optimized UFP-QAPP worksheets, and various tables, figures, and appendices to more fully describe aspects of this drinking water sampling program.

The optimized worksheets reflect the consolidation of several worksheets into a final product containing 27 worksheets. For ease of reference, the revised worksheets are named to reflect the original worksheets on which they are based.

Worksheets

QAPP Worksheet #1 & 2: Title and Approval Page
(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

This worksheet identifies the principal points of contact for all organizations having decision authority in the project and documents their commitment to implement the QAPP.

1. Project Identifying Information
 - a. Site name/project name: Chambers Works/PFAS Drinking Water Sampling Program
 - b. Site location/number: Deepwater, Salem County, New Jersey
 - c. Chemours¹ Project Number: 509026/509048
 - d. AECOM Project Number: 60593793/60595223
2. Lead Organization: Chemours Corporate Remediation Group (CRG)
3. Lead Contractor: AECOM
4. Approvals:

_____ Andrew Hartten, Chemours Project Director	_____ Date
_____ Scott Norcross, AECOM Project Manager	_____ Date
_____ Katherine Davis, AECOM Project Technical Adviser	_____ Date
_____ Kelly Rinehimer, AECOM Project Chemist	_____ Date
_____ Lance Holman, AECOM Quality Assurance (QA) Officer	_____ Date
_____ Eleni Kavvadias, USEPA Region 2	_____ Date

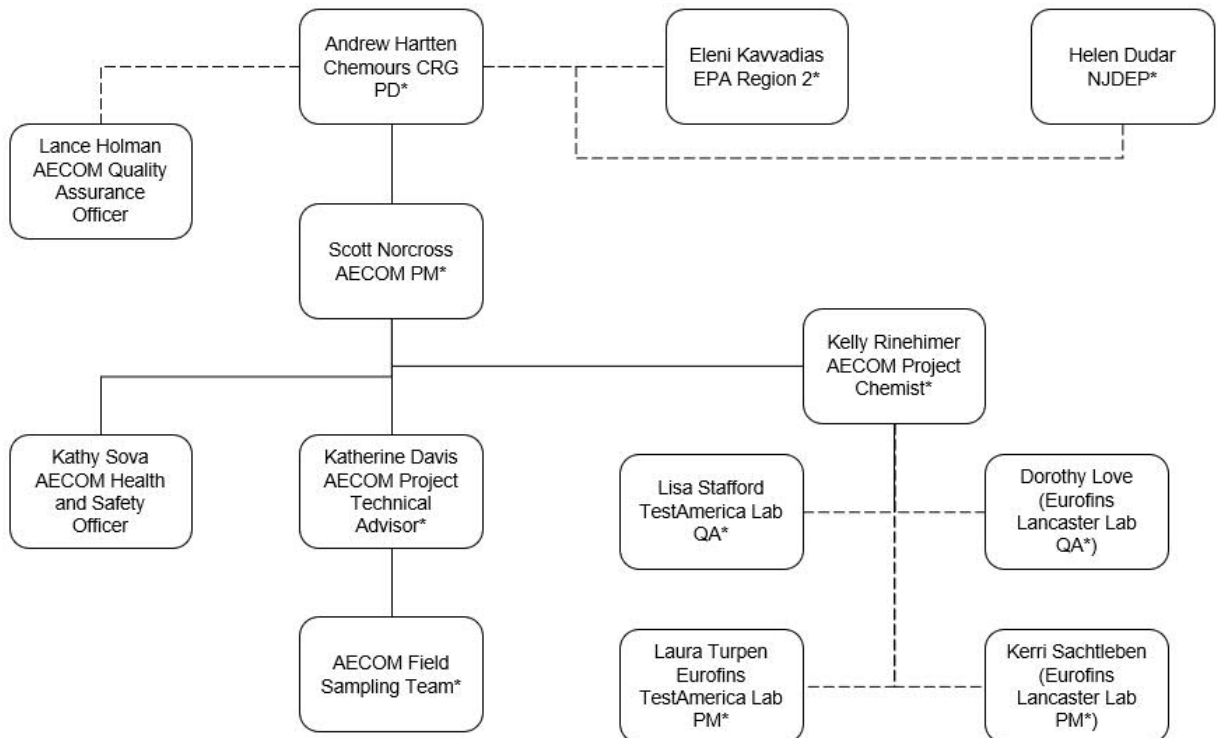
¹ Effective February 1, 2015 the Performance Chemicals reporting segment of E. I. du Pont de Nemours and Company (DuPont) completed a name and ownership change to The Chemours Company FC LLC (Chemours). Chemours operated as a wholly owned subsidiary of DuPont until June 30, 2015. Effective July 1, 2015, Chemours became a wholly independent publicly traded company; therefore, on this date, the site came under the operational control of Chemours.

**QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)**

This worksheet identifies key project personnel, as well as lines of authority and lines of communication among the lead agency, prime contractor, subcontractors, and regulatory agencies.

This worksheet also documents recipients of copies of the QAPP.

*QAPP recipient Lines of authority = single line Lines of Communication = dashed line



**QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet
(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)**

This worksheet is used to identify key project personnel for each organization performing tasks defined in this QAPP.

ORGANIZATION: Chemours

Name	Project Title/Role	Education/ Experience	Specialized Training/Certifications	Signature/Date
Andrew Hartten	Project Director	BS	PFASs Sampling Checklist	

ORGANIZATION: AECOM

Name	Project Title/Role	Education/ Experience	Specialized Training/Certifications	Signature/Date
Scott Norcross	Project Manager	BS	PFASs Sampling Checklist	
Katherine Davis	Project Technical Adviser	PhD	PFASs Sampling Checklist	
Lance Holman	QA Officer	BS		
Kelly Rinehimer	Project Chemist	BS	PFASs Sampling Checklist	

ORGANIZATION: Eurofins Lancaster

Name	Project Title/Role	Education/ Experience	Specialized Training/Certifications	Signature/Date
Dorothy Love	QA Director	B.S., Environmental Health, 38 years' experience		Dorothy Love 7/8/2020
Kerri Sachtleben	Senior Project Manager	BS, Marine Science, 22 years' experience		Kerri Sachtleben 7/8/20

ORGANIZATION: Eurofins TestAmerica Sacramento

Name	Project Title/Role	Education/ Experience	Specialized Training/Certifications	Signature/Date
Lisa Stafford	QA Manager	BS	40-hour Eurofins TestAmerica QA Manager	
Laura Turpen	Project Manager	BS		

*Signatures indicate personnel have read and agree to implement this QAPP as written.

Project roles and responsibilities are described further in the text that follows:

Project Director

Andrew Hartten will be the Chemours project director. The project director's responsibilities will be as follows:

- Provide strategic-level review of technical activities.
- Provide direction involving the drinking water sampling program.
- Approve project-specific procedures and internally prepared plans, drawings, and reports.
- Provide guidance to the project team.
- Act as the Chemours representative for the drinking water sampling program activities to regulators and other stakeholders.

Project Manager

Scott Norcross, AECOM, will act as the project manager for the drinking water sampling program. The project manager will be the primary point of contact with Chemours and will be responsible for all technical, financial, and scheduling matters. The project manager's other responsibilities will be as follows:

- Assign duties to the project team and orienting the team to project needs and requirements.
- Disseminate project-related information from Chemours.
- Act as a liaison with subcontractor organizations (unless specifically delegated to others).
- Interact with the QA officer and health and safety officer to ensure that these programs are functioning effectively and safely.
- Serve as the collection point for project team reporting of nonconformance with QA procedures or changes in project documents and activities.

Health and Safety Officer

Kathy Sova, AECOM, is the health and safety officer for the project. The health and safety officer will be responsible for developing, reviewing, and approving the project health and safety plan (HASP). The health and safety officer will ensure that the project HASP is consistent with applicable state and federal regulations and will be responsible for implementing the HASP.

Project Technical Adviser

Katherine Davis, AECOM, is the project technical adviser. The project technical adviser will be responsible for the following:

- Coordinating or leading the drinking water sampling activities.
- Interacting with the project chemist regarding sampling events.
- Evaluating drinking water data.
- Leading the preparation of reports and documentation.

Technical Consultants

Senior staff members with expertise in the disciplines associated with the drinking water sampling program are available to the project as needed. These individuals will participate in the project as directed by the project manager.

Technical and Support Staff

Individuals in this category will participate in the drinking water sampling program and will be coordinated by the project manager.

Quality Assurance Officer

Lance Holman, AECOM, is the quality assurance (QA) officer for the drinking water sampling program. The QA officer's responsibilities will be as follows:

- Reviewing the QAPP.
- Administering the QAPP.
- Supervising day-to-day QA activities.
- Notifying personnel of nonconformance or changes in procedures.
- Determining the system and performance audit schedules, if required.

Project Chemist

Kelly Rinehimer, AECOM, will be the project chemist. The project chemist will schedule all sample container orders and analytical requests with the laboratory. The project chemist will also be the point of contact between the laboratory and project team. The project chemist will carry out the day to day QA activities. The project chemist will coordinate internal and third-party review of data generated by the laboratory.

Laboratory Personnel

Eurofins Lancaster (ELLE), located in Lancaster, Pennsylvania, is the laboratory that will perform the sample analysis. Eurofins TestAmerica Laboratories (TAL) Sacramento, located in West Sacramento, California, is another laboratory that may also perform sample analysis if ELLE can't accept samples. The regulatory agencies will be notified of any change in the designated laboratories.

The key laboratory personnel for the drinking water sampling program will be the ELLE project manager, Kerri Sachtleben and/or the TAL project manager, Laura Turpen. The analytical laboratory project manager will be responsible for execution of the analytical testing program for the project. The laboratory project manager will be responsible for laboratory analyses and data processing. The laboratory project manager will be the point of contact for the project chemist and QA officer and will be assisted by the laboratory QA director, who is responsible for ensuring that laboratory internal QA procedures are followed and for processing QA data. The laboratory project manager is also responsible for submitting the final data package, including hardcopy deliverable and electronic data deliverable (EDD), within the requested turnaround time.

The laboratories have signed a contract with Chemours detailing the terms and conditions for services. This contract includes a guarantee to dispose of samples following analysis in accordance with all pertinent federal, state, and local laws and ordinances.

**QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)**

This worksheet is used to document specific issues (communication drivers) that will trigger the need to communicate with other project personnel or stakeholders. Its purpose is to ensure there are procedures in place for providing the appropriate notifications and generating the appropriate documentation when handling important communications, including those involving regulatory interfaces, unexpected events, emergencies, non-conformances, and stop-work orders.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory agency interface	Chemours	Andrew Hartten	Andrew.S.Hartten@AECOM.com 302-773-1289	As needed, phone calls and email
Residential referrals to USEPA (Pat Seppi, 646-369-0068) and or NJDEP (Mark Herzburg, 609-633-1369)	AECOM	Shannon Murphy	shannon.murphy@AECOM.com 856-981-1510	As needed; referrals documented in the residential contacts tracking spreadsheets provided to USEPA and NJDEP for the monthly update meetings
Sampling offer letters mailed to residents and copied to USEPA and NJDEP	AECOM	Kathy Davis	Katherine.I.davis@AECOM.com 302-781-5890	PDFs of sampling offer letters are emailed to USEPA and NJDEP
Qualification for well sampling with determination made by speaking with the residents	AECOM	Shannon Murphy/Kathy Davis	shannon.murphy@AECOM.com 856-981-1510 Katherine.I.davis@AECOM.com 302-781-5890	Communication between Shannon and Kathy via phone calls, emails and between Shannon and the residents; information is documented in the residential contact tracking spreadsheet provided to USEPA and NJDEP for the monthly update meetings

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Sample scheduling and sample collection for newly identified drinking-water wells and for the quarterly operation, maintenance and monitoring (OM&M) samples for drinking-water well where treatment has been installed	AECOM	Shannon Murphy/Maddie Reim	shannon.murphy@AECOM.com 856-981-1510 madelene.reim@aecom.com 856-540-2666	Communication between Shannon and Maddie and the residents via phone calls; information is documented in the residential contact tracking spreadsheet provided to USEPA and NJDEP for the monthly update meetings; OM&M sampling documented in the quarterly reports submitted to USEPA and NJDEP within two weeks after the last OM&M samples for that quarter has been finalized
Sample submission/receipt	AECOM/ Eurofins	Shannon Murphy/Maddie Reim/Kelly Rinehimer/Kerri Sachtleben/Laura Turpen	shannon.murphy@AECOM.com 856-981-1510 madelene.reim@aecom.com 856-540-2666 Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Shannon, Maddie, Kelly and Eurofins
Stop work due to safety issues	AECOM	Shannon Murphy/Maddie Reim/ Scott Norcross	shannon.murphy@AECOM.com 856-981-1510 madelene.reim@aecom.com 856-540-2666 Scott.Norcross@AECOM.com 302-547-6569	As needed, phone calls between Shannon, Maddie and Scott; documented in emails to USEPA and NJDEP
Laboratory quality control variances	AECOM/ Eurofins	Kerri Sachtleben/Laura Turpen (or designee) Kelly Rinehimer	Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Kelly and Eurofins

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Analytical corrective actions	AECOM/ Eurofins	Kerri Sachtleben/ Laura Turpen (or designee) Kelly Rinehimer	Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Kelly and Eurofins
Data verification issues, e.g., incomplete records	AECOM/ Eurofins	Kelly Rinehimer/Kerri Sachtleben/ Laura Turpen (or designee)	Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Kelly and Eurofins
Data validation issues, e.g., non-compliance with procedures	AECOM/ Eurofins	Kelly Rinehimer/Kerri Sachtleben/ Laura Turpen (or designee)	Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Kelly and Eurofins
Data review corrective actions	AECOM/ Eurofins	Kelly Rinehimer/Kerri Sachtleben/ Laura Turpen (or designee)	Kelly.Rinehimer@AECOM.com 610-234-4258 KerriSachtleben@eurofinsUS.com 717-556-7376 Laura.Turpen@testamericainc.com 916-374-4414	Communication via emails, phone calls between Kelly and Eurofins
New drinking-water well result letter preparation and transmittal	AECOM	Kathy Davis/Shannon Murphy	Katherine.I.davis@AECOM.com 302-781-5890 shannon.murphy@AECOM.com 856-981-1510	Generate and mail hard copies to residents, generate PDF and email to USEPA, NJDEP, municipal clerks and the Salem County Health Department, phone calls between Kathy and Shannon
OM&M sample result letter preparation and transmittal	AECOM	Kathy Davis/Shannon Murphy	Katherine.I.davis@AECOM.com 302-781-5890 shannon.murphy@AECOM.com 856-981-1510	OM&M result letters generated and mailed to residents in batches after results are finalized, OM&M result letters included in the quarterly reports are submitted to USEPA and NJDEP within two weeks after the last OM&M sample for that quarter has been finalized

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
OM&M Quarterly Reports (includes full data deliverables for OM&M samples)	AECOM	Kathy Davis/Kelly Rinehimer	Katherine.I.davis@AECOM.com 302-781-5890 Kelly.Rinehimer@AECOM.com 610-234-4258	Quarterly OM&M reports will be submitted within two weeks after the last OM&M result for that quarter has been finalized; reports are submitted to USEPA and NJDEP
Full data deliverables for the new drinking-water well sampling	AECOM	Kathy Davis/Kelly Rinehimer	Katherine.I.davis@AECOM.com 302-781-5890 Kelly.Rinehimer@AECOM.com 610-234-4258	Will be submitted semiannually on the last business day of May and November each year to USEPA and NJDEP
Full data deliverables for the Qualification Re-Evaluation Program sampling	AECOM	Kathy Davis/Kelly Rinehimer	Katherine.I.davis@AECOM.com 302-781-5890 Kelly.Rinehimer@AECOM.com 610-234-4258	Will be submitted semiannually on the last business day of May and November each year to USEPA and NJDEP
Ongoing 2016 Residential Drinking-Water Well Surveying and PFAS Sampling Program Update Reports	AECOM	Kathy Davis	Katherine.I.davis@AECOM.com 302-781-5890	At key milestones in the project, summary reports will be generated and submitted to USEPA and NJDEP

**QAPP Worksheet #9: Project Planning Session Summary
(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)**

A copy of this worksheet is completed for each project planning session, whether sessions are internal (project teams only) or external (includes regulators and/or stakeholders). It is used to provide a concise record of participants, key decisions or agreements reached, and action items.

Date of planning session: April 4, 2016
Location: AECOM, 4051 Ogletown Road, Newark, DE 19713
Purpose: Project scoping

Participants:

Name	Organization	Title/Role	Email/Phone
Andrew Hartten	Chemours	Project Director	Andrew.S.Hartten@chemours.com 302-773-1289
Mark Houlday	AECOM	Project Manager	Mark.Houlday@AECOM.com 302-547-6569
Katherine Davis	AECOM	Project Technical Adviser	Katherine.I.davis@AECOM.com 302-781-5890
Mike Aucoin	AECOM	Project Chemist	michael.aucoin@AECOM.com 302-781-5873

Notes/Comments:

Consensus decisions made:

Action Items:

Action	Responsible Party	Due Date
Prepare the QAPP	Mike Aucoin, AECOM	April 15, 2016

Date of planning session: May 12, 2020
Location: AECOM, 4051 Ogletown Road, Newark, DE 19713
Purpose: Discussion of USEPA and NJDEP comments on the January 6, 2020 Chambers Works Residential Program SOPs

Participants:

Name	Organization	Title/Role	Email/Phone
Andrew Hartten	Chemours	Project Director	Andrew.S.Hartten@chemours.com 302-773-1289
Katherine Davis	AECOM	Project Technical Adviser	Katherine.I.davis@AECOM.com 302-781-5890
Eleni Kavvadias	EPA	Life Scientist	kavvadias.eleni@epa.gov 212-637-4138
Helen Dudar	NJDEP	Case Manager	Helen.Dudar@dep.nj.gov 609-633-9279

Notes/Comments:

Consensus decisions made:

Action Items:

Action	Responsible Party	Due Date
Revise the QAPP	Kelly Rinehimer and Katherine Davis, AECOM	July 14, 2020

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

This worksheet is used to present the project's conceptual site model (CSM). The CSM for Poly- and Perfluoroalkyl Substances (PFAS), Chemours Chambers Works, Deep Water, New Jersey, was submitted to USEPA and NJDEP in July 2017. Information from that document is summarized below. Note that the information gathered during the installation and sampling of the off-site monitoring well clusters and the residential sampling program will fill in data gaps and allow for the CSM to be revised as new data become available.

Summary

AECOM, on behalf of the Chemours Company (Chemours), has prepared this Conceptual Site Model (CSM) for Poly- and Perfluoroalkyl Substances (PFAS) for the Chambers Works Complex (the site) located in Deepwater, New Jersey, as requested by New Jersey Department of Environmental Protection (NJDEP) in an April 6, 2016 letter to Chemours. The CSM presented herein was created in general accordance with the NJDEP Technical Guidance for the Preparation and Submission of a CSM (NJDEP, 2011). The areal scope of the CSM includes the Chambers Works Complex, the adjoining Delaware River, and surrounding off-site areas.

The purpose of this CSM is to identify sources of PFAS and potential migration pathways that may have resulted in detections of PFAS in off-site environmental media receptors identified in this CSM as off-site surface water, sediment, and residential well water. As such, this CSM incorporates PFAS data associated with soil, sediment, surface water, groundwater, treatment plant effluent, and stack and vent emissions collected at and around the site since 2003 to construct the CSM framework.

A potential fourth source for PFAS detections should be recognized. This includes consumer and industrial products that contain PFAS, such as windshield wiper fluid, cosmetic products, and fire extinguishers. These products are not associated with the site but are frequently present in homes and businesses and could also contribute to the detections of PFAS in off-site environmental media. Although the possibility of these sources is acknowledged, no measured data were included in the development of this CSM.

Three primary sources of PFAS have been identified at Chambers Works: PFAS [e.g., perfluorooctanoic acid (PFOA)] were used or unintentionally created during the manufacturing of fluoroelastomers and fluorotelomers starting in the 1960s; PFAS were associated with breakdown constituents related to precursor compounds (e.g. fluorotelomer alcohols); and liquid wastes that potentially contained PFAS were brought to Chambers Works for treatment at the site's Wastewater Treatment Plant (WWTP). Since 2003, the use of PFOA has been reduced at Chambers Works. Chambers Works has continued to implement reduction programs that have resulted in an overall 99% reduction in PFOA emissions since 2000.

Migration pathways for the movement of PFAS from sources to off-site environmental media receptors include air emissions and downwind movement of PFAS from stacks and vents during manufacturing processes; discharge of a treated effluent that contains PFAS from the WWTP through two permitted outfalls to the Delaware River; stormwater runoff that contains PFAS and discharges through outfalls to Salem Canal; and to a lesser extent, groundwater containing PFAS that discharges through the shallow aquifer to the Delaware River. However, a sheet pile barrier (SPB) was installed along the Salem Canal, which contains groundwater on-site and limits the discharge from the shallow aquifer to off-site surface water. Discharge will be reduced with the installation of the final section of the SPB engineering control

along the Delaware River in the manufacturing area in 2017. Because groundwater flow is controlled by the site Interceptor Well System (IWS) and the SPB controls groundwater discharge along the southwestern perimeter, there is no migration pathway through groundwater to off-site well locations.

Several investigations have been completed and have adequately characterized PFAS in the media investigated to develop this CSM. PFAS have been detected in soil and groundwater at the Chambers Works Complex. PFAS were detected most frequently and at the highest concentrations in shallow groundwater samples closest to known site process areas that used PFAS. Concentrations decrease with increasing depth and distance from known process buildings.

Detections of PFAS in surface water in the Salem Canal adjacent to the site indicated little difference from upgradient background locations. For the Delaware River, PFAS concentrations were detected in surface water adjacent to the site. For sediment samples collected from the Delaware River and Salem Canal, higher PFAS detections were noted in samples collected near stormwater and permitted effluent discharge locations.

Off-site groundwater investigations in 2007, 2009, 2010, 2014, and 2016, as well as the Ongoing Residential Drinking-Water Well Surveying and PFAS Sampling Program, indicate the presence of PFAS in off-site groundwater, at times exceeding NJDEP Drinking Water Standards for PFOA (0.014 ug/L), perfluorooctane sulfonate [(PFOS); (0.013 ug/L)], or perfluorononanoic acid [(PFNA); (0.013 ug/L)]. While air emissions from the site contribute to these detections, off-site use of PFAS-containing products unrelated to the site may also add to these detections as the variability in PFAS constituents detected and the variable nature of the observed concentrations does not support a single point of origin in all cases. However, Chemours is actively working with NJDEP and USEPA to continue to investigate and to address potential drinking water exposure by offering to treat off-site drinking water for PFAS, if criteria are exceeded.

QAPP Worksheet #11: Project/Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

This worksheet is used to develop and document data quality objectives (DQOs) using USEPA's 7-step DQO process.

1. State the Problem – Chemours has agreed to perform additional sampling and analysis of residential drinking water from homes located within approximately two miles of the Chambers Works site boundary. Only wells permitted as potable wells are eligible for inclusion in this program.
2. Identify the Goals of the Study – The goals of the study are to further delineate the nature and extent of PFAS contamination by sampling or resampling residential drinking water sources within approximately two miles of the Chambers Works site and by laboratory analysis of an expanded target list of compounds. In addition, if PFOA concentrations in those wells exceed the New Jersey drinking water standards for PFOA, PFOS or PFNA, installation of residential granular activated carbon (GAC) treatment (or an alternative treatment approved by USEPA and NJDEP) will be offered at no cost to the property owner.
3. Identify Information Inputs – An existing database of residential drinking water sources within approximately two miles of the Chambers Works site boundary will be supplemented as necessary to generate a final list of residential drinking-water sources that will be offered sampling or resampling. The current investigation area has expanded beyond the two-mile radius based on the results for drinking-water wells sampled. Analytical results will supplement understanding of any PFAS contaminant migration outside of the Chambers Works Site.
4. Define the Boundaries of the Study – Chemours initially agreed to perform sampling and resampling of residential drinking water sources within approximately two miles of the Chambers Works Site. Based on results, the study area has and continues to expand.
5. Develop the Analytic Approach – Laboratories providing analytical testing will be certified by NJDEP for the matrix and parameter. The results of the sampling or resampling will be summarized in a results table and may be mapped to display the nature and extent of contaminant migration. Drinking water samples will be analyzed by USEPA Method 537.1 to reach a reporting limit of 2 ng/L, as communicated to Chemours by USEPA Region 2 and if achievable for each of the following target compounds¹:

CAS Num	Compound Abbreviation	Analyte
958445-44-8	ADONA	4,8-Dioxa-3H-perfluorononanoic acid

¹ Note that if a drinking-water well exceeds the NJDEP Drinking Water Standards (see Worksheet #10) for either PFOA, PFOS or PFNA and a treatment system is installed, samples collected for the quarterly operation, maintenance, and monitoring program will be analyzed by a New Jersey certified lab using USEPA Method 537.1 for PFOA, PFOS and PFNA only. In addition, if a drinking water does not exceeds the NJDEP Drinking Water Standards (see Worksheet #10) for either PFOA, PFOS or PFNA, then that well is included in the Qualification Re-Evaluation Program which was implemented December 1, 2020 and follow up sampling events for this program will include analysis by a New Jersey certified lab using USEPA Method 537.1 for PFOA, PFOS and PFNA only.

CAS Num	Compound Abbreviation	Analyte
756426-58-1	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid
763051-92-9	11Cl-F3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
13252-13-6	HFPO-DA	Hexafluoropropylene oxide dimer acid
375-73-5	PFBS	Perfluorobutanesulfonic acid
335-76-2	PFDA	Perfluorodecanoic acid
307-55-1	PFDoA	Perfluorododecanoic acid
375-85-9	PFHpA	Perfluoroheptanoic acid
355-46-4	PFHxS	Perfluorohexanesulfonic acid
307-24-4	PFHxA	Perfluorohexanoic acid
375-95-1	PFNA	Perfluorononanoic acid
1763-23-1	PFOS	Perfluorooctanesulfonic acid
335-67-1	PFOA	Perfluorooctanoic acid
376-06-7	PFTeA	Perfluorotetradecanoic acid
72629-94-8	PFTrDA	Perfluorotridecanoic acid
2058-94-8	PFUnA	Perfluoroundecanoic acid
2991-50-6	NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid
2355-31-9	NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid

6. Specify Performance or Acceptance Criteria – Laboratory QC limits of accuracy and precision for each target compound can be found in Table 1 and will be used to judge the usability of the analytical data for project purposes. Further evaluation of the data will reflect blank contamination, if any, adherence to established hold times, and accuracy and precision results for field QC samples.
7. Develop the Detailed Plan for Obtaining Data – The basis for the sampling design rests on the results of previous sampling efforts, as well as identification of further residential water sources within defined boundaries outside of the Chambers Works, as described in Worksheet #17.

**QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)**

This worksheet documents the quantitative measurement performance criteria (MPC) in terms of precision, bias, and sensitivity for both field and laboratory measurements and is used to guide the selection of appropriate measurement techniques and analytical methods. MPC are developed to ensure collected data will satisfy the PQOs or DQOs documented on Worksheet #11.

Matrix: Drinking Water
Analytical Group or Method: PFAS/USEPA Method 537.1
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	RPD \leq 30% when PFASs are detected in both samples \geq 5X sample-specific RL. Difference < sample specific RL when one or both samples < 5X sample specific RL
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD \leq 30%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	70-130%
Analytical Accuracy/Bias (laboratory)	Low Fortified Laboratory Control Samples	50-150%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Samples	70-130%
Analytical Accuracy/Bias (matrix interference)	Low Fortified Matrix Spike Samples	50-150%
Analytical Precision (matrix interference)	Matrix Spike Duplicates	RPD \leq 30%
Analytical Precision (matrix interference)	Low Fortified Matrix Spike Duplicates	RPD \leq 50%
Analytical Accuracy/Bias (matrix interference)	Internal Standard Analytes	The IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration and must not deviate by more than 70-140% from the most recent CCV standard
Overall accuracy/bias (contamination)	Field Reagent Blanks (FRB)	No target analyte concentrations > RL
Analytical Accuracy/Bias (matrix interference)	Surrogate Analytes	70-130%

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Completeness	Percentage of valid data compared to total data collected	100% ¹

¹ 100% of the residential well locations identified under this program will be contacted and will be sampled if the residence responds and the well qualifies for sampling or resampling; individual residences will be contacted and offered resampling if data review or validation judges laboratory results to be invalid.

QAPP Worksheet #13: Secondary Data Uses and Limitations
(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements For Evaluating Existing Data)

This worksheet is used to identify sources of secondary data (i.e., data generated for purposes other than this specific project or data pertinent to this project generated under a separate QAPP) and summarize information relevant to their uses for the current project.

Data type	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Analytical	Site files, Locus EIM database	Estimation of sampling scope	Historical off site PFAS data generally limited to PFOA

**QAPP Worksheet #14/16: Project Tasks & Schedule
(UFP-QAPP Manual Section 2.8.2)
(EPA 2106-G-05 Section 2.2.4)**

The following is a project schedule showing specific tasks, the person or group responsible for their execution, and planned start and end dates. The information below is from the June 2016 QAPP for this project. Note that all the activities listed below, are sequential and ongoing with the expansion of the program beyond the two mile-radius.

Due to the expansion of the project over time based on analytical results, the activities listed below are all still ongoing. However, the planned start date, the planned completion date, and deliverable due dates are dependent upon the current phase of the investigation underway. See QAPP Worksheet #6: Communication Pathways for additional timing information. In addition, standard operating procedures for this ongoing program (including for the activities described below) are provided in the "Standard Operating Procedures for the Ongoing 2016 Residential Drinking-Water Well Surveying and PFAS Sampling Program Revision 3" (revised in November 2020 and approved December 1, 2020).

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Mailing of Residential Letters	AECOM	Ongoing	Ongoing	Letters mailed to residents	TBD as new investigation areas are defined
Residential contacts spreadsheet and Sampling Qualification Determination	AECOM, USEPA if residents have questions and the call center refers them to the USEPA contact	Ongoing	Ongoing	Summary spreadsheet of call center contacts; complete spreadsheet will be provided in the final project report	TBD as new investigation areas are defined
Drinking-Water Well Sampling	AECOM	Ongoing	Ongoing	Field notes	TBD as new investigation areas are defined
Analysis	Eurofins Lancaster and/or Eurofins TestAmerica Sacramento	Ongoing	Ongoing	Report of Analyses/Data package	TBD as new investigation areas are defined
Validation - DVM	AECOM	Ongoing	Ongoing	Validation Summary report	TBD as new investigation areas are defined
Validation – third party (10%, see Worksheet 36)	Environmental Standards	Ongoing	Ongoing	Validation Summary report	TBD as new investigation areas are defined
Usability assessment	AECOM	Ongoing	Ongoing	Usability assessment summary report	TBD as new investigation areas are defined

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Results to USEPA, NJDEP, municipal clerks and the Salem County Health Department	AECOM	Ongoing	Ongoing	Spreadsheet and PDFs	TBD as new investigation areas are defined
Result letter to Resident	AECOM	Ongoing	Ongoing	Result Letter	TBD as new investigation areas are defined
Monthly Status Meetings	AECOM	Ongoing	Ongoing	Residential tracking spreadsheets, results mailed spreadsheets	TBD as new investigation areas are defined
Following up with returned letters and residents that are non-responsive to the sampling offer	AECOM/EPA	Ongoing	Ongoing	Door to door contacts and follow up letters and tracking via a public database tracking service	TBD as new investigation areas are defined
Final Project Report generation	AECOM	Ongoing	Ongoing	Final Project Report	TBD as new investigation areas are defined
Quarterly Operation, Maintenance and Monitoring Program for all wells with treatment systems installed	AECOM	Ongoing	Ongoing	OM&M Quarterly Reports (includes full data deliverables for OM&M samples)	Each quarter with the OM&M report due two weeks after the last data are finalized for the quarter.

**QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)**

This worksheet provides project action limits and laboratory-specific detection/quantitation limits for the PFAS Drinking Water Sampling Program:

Matrix: Drinking Water

Analytical Method: PFAS/USEPA Method 537.1

Concentration level (if applicable): Low

Laboratory: Eurofins TestAmerica Sacramento and Eurofins Lancaster

Analyte	Project Action Limit (PAL)	PAL Reference	Project Quantitation Limit Goal	Laboratory-specific quantitation limit ⁵	Laboratory-specific detection limit ⁶
PFOA	14 ng/L	NJ Drinking Water Standard ¹	2 ng/L	2 ng/L	0.5 ng/L
PFOS	13 ng/L	NJ Drinking Water Standard ²	2 ng/L	2 ng/L	0.5 ng/L
PFNA	13 ng/L	NJ Drinking Water Standard ³	2 ng/L	2 ng/L	0.5 ng/L
PFASs (other compounds – see Worksheet #11)	2 ng/L	Communication, USEPA Region 2 to Chemours ⁴	2 ng/L	2 ng/L	0.5 ng/L

¹ NJ Drinking Water Standard for Perfluorooctanoic Acid (PFOA)

² NJ Drinking Water Standard for Perfluorooctane Sulfonate (PFOS)

³ NJ Drinking Water Standard for Perfluorononanoic acid (PFNA)

⁴ The PAL for Perfluoroalkyl Substances (PFASs) other than those listed are not true advisory levels but rather reflect the Project Quantitation Limit Goal provided in a communication from USEPA Region 2 to Chemours.

⁵ Laboratory-specific quantitation limit is the laboratory's reporting limit

⁶ Laboratory-specific detection limit is the laboratory's method detection limit

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

This worksheet is used to describe the sampling design and the basis for its selection and documents the last step of the planning process.

Sampling will be offered to owners of all drinking-water wells which have been permitted as Category 1 Potable Water Supply Wells. Lists of residents to be included in a specific phase of the ongoing 2016 program are developed via several methods including searches of a tax information database (http://tax1.co.monmouth.nj.us/cgi-bin/prc6.cgi?&ms_user=monm&passwd=data&srch_type=1&adv=2&out_type=2&district=1700), a GIS mapping database (NJ GeoWeb: <https://www.nj.gov/dep/gis/geoweb splash.htm>), a well search database (NJDEP DataMiner: <https://www13.state.nj.us/DataMiner>), and/or field reconnaissance by Chemours representatives. In some situations, specific resident address lists or potential drinking-water well location address lists were or may be provided to Chemours by USEPA or NJDEP. In addition, new well permits are tracked using publicly available databases and/or a publicly available database tracking service and offer letters are mailed to residents or property owners offering sampling of drinking-water wells.

Each resident included in the current phase of the ongoing 2016 program is contacted and offered sampling of their drinking-water well via mailing of a series of three drinking-water well sampling offer letters three weeks apart. PDFs of offer letters are shared with USEPA and NJDEP. In July 2019, Chemours implemented a new process of adding a self-addressed, stamped, response postcard with each offer letter mailed. The response postcard has several check boxes that encourage the resident to request sampling or otherwise share the reason that the offer is not being accepted. If tax records indicate that the property owner does not live where the potential drinking-water well is located, a duplicate letter is mailed to the property owner's address that includes the potential drinking-water well address in the subject line to indicate that sampling is being offered. All outreach efforts for residents and property owners are tracked in the residential contacts spreadsheet that is used to document responses to offer letters and sampling completed. The evergreen residential contacts spreadsheet is provided to USEPA and NJDEP on an approximate monthly basis, prior to project status conference calls. Note that either the resident or the property owner may request sampling of the drinking-water well. The drinking-water well will also be sampled if the resident requests sampling, but the owner does not or is non-responsive to the sampling offer. Additional details of the SOPs used in development of a potential drinking-water well address list and offering of surveying and sampling through submission of the result letter where sampling is completed is provided in Section 3.0 of the Standard Operating Procedures for the Ongoing 2016 Residential Drinking-Water Well Surveying and PFAS Sampling Program (revised in November 2020 and approved December 1, 2020).

If a drinking-water well exceeds the NJDEP Drinking Water Standards (see Worksheet #10) for either PFOA, PFOS or PFNA and a treatment system is installed, the drinking-water well will be pulled into Chemours quarterly OM&M program. Samples collected for the quarterly OM&M program will only be analyzed for PFOA, PFOS and PFNA only by USEPA Method 537.1. Additional details on treatment installation and the quarterly OM&M program are provided in Sections 4 and 5 of the Standard Operating Procedures for the Ongoing 2016 Residential Drinking-Water Well Surveying and PFAS Sampling Program (revised in November 2020 and approved December 1, 2020). Other treatment options include permanent bottled water or, where practical, connection to a public water supply and abandonment (sealing) of the potable well to be protective of the homeowner and reduce the risk of the well being reconnected for residential use.

If a drinking-water well does not exceed the NJDEP Drinking Water Standards (see Worksheet #10) for either PFOA, PFOS or PFNA, then it is automatically included in the Qualification Re-Evaluation Program.

This includes wells that had results below the reporting limit of 0.002 µg/L. This program provides follow-up monitoring of the concentration of PFOA, PFOS, and PFNA in the drinking-water wells over time. The program includes a sequential series of two annual events and three biennial events. Drinking-water wells with concentrations of PFOA, PFOS, or PFNA that do not exceed the evaluation criteria after the completion of this series of monitoring events will be discussed with USEPA and NJDEP to determine if additional monitoring is warranted or not. Additional details on the Qualification Re-Evaluation Program are provided in Section 6 of the Standard Operating Procedures for the Ongoing 2016 Residential Drinking-Water Well Surveying and PFAS Sampling Program (revised in November 2020 and approved December 1, 2020).

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

This worksheet provides a summary of sample types to be collected during the PFAS drinking water sampling program:

Sample ID	Matrix¹	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CWK-D-ADDRESS- Date sampled	DW	Field sample	PFASs	QAPP (see Worksheet 21)	New locations
CWK-D-ADDRESS- Date sampled	DW	Field sample	PFASs	QAPP (see Worksheet 21)	Repeat locations
CWK-D-ADDRESS- Date sampled-D	DW	Field duplicate	PFASs	QAPP (see Worksheet 21)	All locations
FRB-Date sampled	W	Field reagent blank (FRB)	PFASs	QAPP (see Worksheet 21)	Field reagent blank is related to all locations collected and shipped in a batch representing up to 2 consecutive field days OR up to 20 samples, whichever is fewer FRBs.
CWK-D-ADDRESS- Date sampled-PRE	DW	Field sample	PFASs	QAPP (see Worksheet 21)	Locations with pre- existing treatments
CWK-D-ADDRESS- Date sampled-POST	DW	Field sample	PFASs	QAPP (see Worksheet 21)	Locations with pre- existing treatments
C8QQYY-ADDRESS- PT, -BED1, -BED2	DW	Field sample	PFASs	QAPP (see Worksheet 21)	GAC Locations

¹ Key: DW = drinking water, W = water

**QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times
(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)**

The purpose of this worksheet is to serve as a reference guide for field personnel. It is also an aid to completing the Chain of Custody form and shipping documents.

Laboratory (Name, sample receipt address, POC, e-mail, and phone numbers): Eurofins Lancaster Laboratories
2425 New Holland Pike
Lancaster, PA 17601
Kerri Sachtleben
Tel: 717-556-7376

List any required accreditations/certifications: NJDEP
Sample Delivery Method: Overnight courier such as FedEx

Analyte/ Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround	Data Validation Turnaround
PFASs	Drinking Water	USEPA 537.1 T-PFAS- WI25232 (Appendix A)	6/30/2021	2 x 250 mL HDPE Bottles	Trizma; at or below 10°C	14 days	28 days	21 days	30 days

Laboratory (Name, sample receipt address, POC, e-mail, and phone numbers):

Eurofins TestAmerica
880 Riverside Parkway
West Sacramento, CA 95605
Laura Turpen
Laura.Turpen@testamericainc.com
Tel: 916-374-4414
Fax: 916-372-1059

List any required accreditations/certifications: NJDEP

Sample Delivery Method: Overnight courier such as FedEx

Analyte/ Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround	Data Validation Turnaround
PFASs	Drinking Water	USEPA 537.1 WS-DW-0004 Rev. 2.7 (Appendix A)	6/30/2021	2 x 250 mL HDPE Bottles	Trizma; at or below 10°C	14 days	28 days	21 days	30 days

QAPP Worksheet #20: Field QC Summary
(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

This worksheet provides a summary of the types of field samples and associated QC samples to be collected and analyzed for the project.

This project is ongoing and continues to expand and therefore the estimated maximum number of samples is unknown. The information in the table below provides an estimated summary of the QC samples to be collected and analyzed per 100 field samples collected for the project.

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Reagent Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses¹
Drinking Water	PFASs	100	5	5	5	5	0	0	N/A	120

¹ All numbers of samples, including the total number of analyses, reflect the estimated maximum possible samples based on the known and estimated number of residential drinking water samples present in the study area.

**QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)**

This worksheet is intended to document the specific field sampling procedures being implemented, which is important for measurement traceability and to avoid contamination of samples that may bias the sample results.

PFASs present extraordinary opportunities for contamination during sampling and therefore sampling personnel will review the PFASs Sampling Checklist document (Appendix B) and follow the recommended precautions found in that document during drinking water sampling.

Information related to collection of each drinking-water sample will be recorded on a data collection sheet (Appendix C) in a sampling book with carbon copy sheets.

Drinking water will be sampled from taps at residential locations. All taps will be flushed for two minutes prior to collecting the water sample. Residential wells that are not used on a routine basis will be purged to remove two to three pressure tank volumes to provide a representative groundwater sample. The following procedure will be followed during sampling from taps:

1. Locate an appropriate tap water source, typically the kitchen tap. If there is a pre-existing treatment system (other than a water softener system), a prior to treatment ("Pre") and an after treatment ("Post") sample will be collected.
2. Open the cold valve and allow water to run for at least two minutes to flush the valve system and supply lines.
3. Remove the bottle cap, place the bottle under the tap, and fill. If the bottle will not fit under the tap faucet, then look for another appropriate source. Do not use a secondary container to fill the bottle.
4. Recap the sample bottle.
5. Affix a sample label, unless the label was affixed by the laboratory.
6. Place the sample in a cooler of ice.
7. Complete the COC form.

For the OM&M program, samples will be collected from sampling ports on the treatment system using the following procedure:

1. If the system is bypassed upon arrival, flush system for 10 minutes from port three prior to collecting samples.
2. The sample from port three (after BED2) is collected first followed by the sample from port two (after BED1). During the third quarter of the year a sample from port one will be collected and called the PT, or Prior to Treatment sample.
3. Remove the bottle cap, place the bottle under the tap, and fill.
4. Recap the sample bottle.
5. Affix a sample label, unless the label was affixed by the laboratory.
6. Place the sample in a cooler of ice.
7. Complete the COC form.

To ensure against cross-contamination between drinking water sampling locations, the sampler collecting the samples will wear clean, disposable latex and/or nitrile gloves and limit his/her contact with the samples. Sample bottles and containers will be prepared by the contracted laboratory and will be sealed to ensure cleanliness. Sample bottles will not be cleaned or reused in the field.

A field reagent blank (FRB) will be handled along with each sample set. Field reagent blank is related to all locations collected and shipped in a batch representing up to 2 consecutive field days OR up to 20 samples, whichever is fewer FRBs.

Laboratory personnel will fill the field blank sample bottle with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) will also be shipped. At the sampling site (CWK), the sampler must open the shipped FRB and pour the preserved reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAS were not introduced into the sample during sample collection/handling.

The same batch of preservative must be used for the FRBs as for the field samples.

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field equipment is not anticipated to be used during residential drinking water sampling.

**QAPP Worksheet #23: Analytical SOPs
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)**

This worksheet documents information about the specific sample preparation and analytical procedures to be used, which is important for measurement traceability.

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
T-PFAS-WI25232	Perfluorinated Alkyl Substances (PFASs) in Drinking Water by Method 537.1 Version 1.0	Definitive	Drinking Water/PFASs	NA	N
WS-DW-0004 Rev. 2.7	Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction (SPE) and Analysis by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS) [Methods 537 and 537.1]	Definitive	Drinking Water/PFASs	NA	N

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

This worksheet is completed for the analytical instruments used in the laboratory.

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
LC/MS/MS	Minimum five-point initial calibration for average response factor and linear fit; six points are required for quadratic fit, lowest concentration standard at or below the reporting limit	0.25 – 50 ng/mL (upper levels may not be included in order to improve calibration fit)	Initial calibration prior to sample analysis	Lowest calibration standard (<RL) \pm 50% of true value. Calibration standards > RL \pm 30% of true value	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration	Lab Manager/Analyst ¹	WS-DW-0004 Rev. 2.7
LC/MS/MS	Second-source calibration verification	Mid-range of calibration curve	Once per initial calibration, following initial calibration	$\%D \pm 30\%$ of true value	Rerun the Initial Calibration Verification (ICV). Remake or acquire a new ICV. Evaluate the instrument conditions. Evaluate the initial calibration standards	Lab Manager/Analyst ¹	WS-DW-0004 Rev. 2.7

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
LC/MS/MS	Daily calibration verification	Per SOP	Before sample analysis, after every 10 field samples, and at the end of the sequence	%D \pm 50% of true value for CCV < RL %D \pm 30% of true value for CCV > RL	Inject another Continuing Calibration Verification (CCV) standard. If still out of control then there may be a problem with the standards, the instrument, or the column. If the column is replaced, then a new curve is necessary	Lab Manager/Analyst ¹	WS-DW-0004 Rev. 2.7
LC/MS/MS	Minimum five-point initial calibration for average response factor and linear fit; six points are required for quadratic fit, lowest concentration standard at or below the reporting limit	0.25 – 50 ng/mL (upper levels may not be included in order to improve calibration fit)	Initial calibration prior to sample analysis	Lowest calibration standard (<RL) \pm 50% of true value. Calibration standards > RL \pm 30% of true value	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration	Lab Manager/Analyst ¹	T-PFAS-WI25232

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
LC/MS/MS	Second-source calibration verification	Mid-range of calibration curve.	Once per initial calibration, following initial calibration	%D \pm 30% of true value	Rerun the ICV. Remake or acquire a new ICV. Evaluate the instrument conditions. Evaluate the initial calibration standards	Lab Manager/Analyst ¹	T-PFAS-WI25232
LC/MS/MS	Daily calibration verification	Per SOP	Before sample analysis, after every 10 field samples, and at the end of the sequence	%D \pm 50% of true value for CCV < RL %D \pm 30% of true value for CCV > RL	Samples that are not bracketed by acceptable CCV runs must be reanalyzed	Lab Manager/Analyst ¹	T-PFAS-WI25232

¹ The analyst initiates the corrective action and the lab manager and analyst are responsible for the corrective action.

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

This worksheet provides a summary of procedures for analytical instrument maintenance, testing, and inspection.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
LC/MS/MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Eurofins TestAmerica Chemist	WS-DW-0004 Rev. 2.7
LC/MS/MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Eurofins Lancaster Chemist	T-PFAS-WI25232

**QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal
(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)**

This worksheet is used to document responsibilities for maintaining custody of samples from sample collection through disposal.

Sampling Organization: AECOM

Laboratory: Eurofins Lancaster and/or Eurofins TestAmerica Sacramento

Method of sample delivery (shipper/carrier): FedEx priority overnight

Number of days from reporting until sample disposal: The laboratory will hold samples for 30 days beyond reporting, or longer if space allows.

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	AECOM, Field Personnel	CRG Chain of Custody SOP (Appendix D)
Chain-of-custody form completion	AECOM, Field Personnel	CRG Chain of Custody SOP (Appendix D)
Packaging	AECOM, Field Personnel	Shipping SOP (Appendix E)
Shipping coordination	AECOM, Field Personnel	Shipping SOP (Appendix E)
Sample receipt, inspection, & log-in	Eurofins Lancaster, Sample Receiving Technician	S-SA-WI10723, S-SA-WI10725 and S-SA-WI10743 (Appendix F)
Sample custody and storage	Eurofins Lancaster, Sample Receiving Technician	QA-SOP11914 and S-SS-WI12042 (Appendix F)
Sample disposal	Eurofins Lancaster, Sample Receiving Technician	S-SS-WI12042 (Appendix G)
Sample receipt, inspection, & log-in	Eurofins TestAmerica, Sample Receiving Technician	WS-QA-0003 (Appendix F)
Sample custody and storage	Eurofins TestAmerica, Sample Receiving Technician	WS-QA-0003 (Appendix F)
Sample disposal	Eurofins TestAmerica, Waste Disposal Technician	WS-EHS-0001 (Appendix G)

**QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)**

The purpose of this worksheet is to ensure that the selected analytical methods are capable of meeting project-specific MPC, which are based on the DQOs.

Matrix: Drinking Water

Analytical Group: PFASs

Analytical Method/SOPs: USEPA Method 537.1 /SOP WS-DW-0004, Revision 2.7 and T-PFAS-WI25232 Revision 3

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per preparatory batch of up to 20 samples	No analytes detected > 1/3 method reporting limit (MRL),	Reprep and reanalyze the method blank and all samples processed with the contaminated blank if detections in the project samples. If problem persists, call PM	Lab Manager / Analyst	All analytes in the method blank must be less than the RL
LCS containing all analytes	One per preparatory batch of up to 20 samples. Perform LCS/LCSD if MS/MSD not submitted.	70-130% RPD ≤ 30%. 50-150% for Low Fortified blank, RPD ≤ 50%	Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for failing compounds, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available	Lab Manager / Analyst	All analytes are evaluated against 70-130%; RPD ≤ 30% or 50-150% (low fortified bank) RPD ≤ 50%

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Internal Standards	Every sample, spiked sample, standard, and method blank	% recovery for each IS in the original sample (prior to dilutions) must not deviate by more than 50% from the average response (peak area) of the initial calibration and must not deviate by more than 70-140% from the most recent CCV standard	Reanalyze in a new capped autosampler vial. If reinjected aliquot produces acceptable response, report results from reinjection. If fails again, analyze the most recent acceptable CCV. If CCV fails, recalibrate the instrument. If CCV passes, re-extract sample. If outside of hold time, contact the client	Lab Manager / Analyst	All analytes are evaluated against the criteria in Table 2
MS/MSD for all analytes	One MS/MSD pair per preparation batch (if sufficient sample provided)	70-130%, RPD \leq 30%. 50-150% for Low Level Spike, RPD \leq 50%	Evaluate the data. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS	Lab Manager / Analyst	All analytes are evaluated against the criteria in Table 1

**QAPP Worksheet #29: Project Documents and Records
(EPA 2106-G-05 Section 2.2.8)
(UFP-QAPP Manual Section 3.5.1)**

This worksheet is used to record information for all documents and records that will be generated for the project. It describes how information will be collected, verified, and stored. Its purpose is to support data completeness, data integrity, and ease of retrieval.

Sample Collection and Field Records			
Record	Generation	Verification	Storage location/archival
Field logbook or data collection sheets (Appendix C)	Shannon Murphy/Maddie Reim	Project Manager (Scott Norcross)	Project File
Chain-of-Custody Forms	Shannon Murphy/Maddie Reim	Project Manager (Scott Norcross)	Project File
Air Bills	Shannon Murphy/Maddie Reim	Project Manager (Scott Norcross)	Project File
Deviations	Shannon Murphy/Maddie Reim	Project Manager (Scott Norcross)	Project File
Corrective Action Reports	Shannon Murphy/Maddie Reim	Project Manager (Scott Norcross)	Project File
Correspondence	Project Technical Adviser (Kathy Davis)	Project Manager (Scott Norcross)	Project File
Resident Database/Spreadsheet	Project Technical Adviser (Kathy Davis)	Project Manager (Scott Norcross)	Project File
Monthly Status Reports	Project Technical Adviser (Kathy Davis)	Project Manager (Scott Norcross)	Project File

Project Assessments			
Record	Generation	Verification	Storage location/archival
Data verification checklists	Project Chemist (Kelly Rinehimer)	Project QA Officer (Lance Holman)	Project File
Data validation report	Project Chemist (Kelly Rinehimer) and Environmental Standards, Inc. (tbd)	Project QA Officer (Lance Holman)	Project File

Project Assessments			
Record	Generation	Verification	Storage location/archival
Data usability assessment report	Project Chemist (Kelly Rinehimer)	Project QA Officer (Lance Holman)	Project File

Laboratory Records			
Record	Generation	Verification	Storage location/archival
Analytical Laboratory Data Packages	Laboratory	Project Chemist/AECOM Team	AECOM project files Laboratory maintains records in accordance with the QAM requirements.
Electronic Data Deliverables	Laboratory	Project Chemist/AECOM Team	AECOM project files Laboratory maintains records in accordance with the QAM requirements

Laboratory Data Deliverables						
Record	PFASs					
Narrative	X					
COC	X					
Summary Results	X					
QC Results	X					
Chromatograms	X					

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action
(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

This worksheet is used to document responsibilities for conducting project assessments, responding to assessment findings and implementing corrective action.

Internal audits and assessments will be performed by the organization primarily responsible for conducting the task being audited. For example, Chemours CRG or its contractor, AECOM, may conduct an assessment of field sample collection activities and the contract laboratory will perform internal audits. Internal audits and assessments for the drinking water program will be conducted as described in this QAPP unless otherwise noted in the individual site work plans.

An example audit checklist is presented as Figure 1. When a significant condition adverse to quality is noted in the field, in the laboratory, or at the office, the cause of the condition will be determined, and corrective action will be initiated by the QA officer to preclude repetition. The nature and cause of the condition, reference documents, and planned corrective actions will be documented and reported to the field team leader, project manager, QA officer, and involved subcontractor management, as appropriate. Implementation of the corrective action will be documented by the QA officer. All project personnel are responsible, as part of their standard work duties, to promptly identify and report conditions adverse to quality and implement the appropriate corrective action.

A corrective action request (CAR), presented as Figure 2, will be used to identify the adverse condition, the reference document(s), and the recommendation of corrective action(s) to be implemented. The CAR will be sent to the person responsible for the item or activity requiring action. The individual receiving the CAR will implement the recommended corrective action of an equivalent corrective action and return the completed form promptly to the QA/QC officer after affixing his/her signature and date. The QA officer will maintain a status control log of CARs and responses, confirm the adequacy of the intended corrective action, and verify implementation of the corrective action. At a minimum, the QA officer will issue and distribute CARs to the originator, project manager, and involved personnel (including subcontractors). CARs will be maintained in the project file.

It will be the project manager's overall responsibility to ensure that all corrective actions are acted upon promptly and satisfactorily.

A technical systems audit of field activities is an on-site, qualitative review of the sampling system to ensure that the activity is being performed in compliance with this QAPP. A technical systems audit of field sampling activities is not planned for this project.

The contract laboratories will be accredited by the state agencies and others, as appropriate. A technical systems audit of the laboratory is not planned for this project.

QAPP Worksheet #34: Data Verification and Validation Inputs
(UFP-QAPP Manual Section 5.2.1 and Table 9)
(EPA 2106-G-05 Section 2.5.1)

This worksheet is used to list the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. Data verification is a check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs and the QAPP. Records subject to verification and validation are listed below.

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	X	
2	Contract	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
Field Records			
5	Field logbooks	X	X
6	Equipment calibration records	X	X
7	Chain-of-Custody Forms	X	X
8	Sampling diagrams/surveys	X	X
9	Relevant Correspondence	X	X
10	Change orders/deviations	X	X
11	Field audit reports	X	X
12	Field corrective action reports	X	X
Analytical Data Package			
13	Cover sheet (laboratory identifying information)	X	X
14	Case narrative	X	X
15	Internal laboratory chain-of-custody	X	X
16	Sample receipt records	X	X
17	Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	X	X
18	Communication records	X	X
19	MDL/RL establishment and verification	X	X
20	Standards Traceability	X	X
21	Instrument calibration records	X	X
22	Definition of laboratory qualifiers	X	X
23	Results reporting forms	X	X
24	QC sample results	X	X
25	Corrective action reports	X	X
26	Raw data	X	X
27	Electronic data deliverable	X	X

**QAPP Worksheet #35: Data Verification Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)**

This worksheet documents procedures that will be used to verify project data. Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete.

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbook	QAPP	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that weather conditions were observed and recorded for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements	Daily - Project Manager At conclusion of field activities - Project QA Officer
Chain-of-custody forms	QAPP, Chain-of-Custody SOP	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservations have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors	Daily - Field Team Leader At conclusion of field activities - Project Chemist
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present	Before release – Laboratory QAM Upon receipt - Project Chemist

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan	Project QA Officer

**QAPP Worksheet #36: Data Validation Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)**

This worksheet documents procedures that will be used to validate project data. Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods/SOPs, and MPC.

Data Verification: AECOM

Data verification is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate.

All data will be provided to the Chemours contactor AECOM in a data package by the laboratory. The data package contains raw data and will be reviewed by the in-house Analytical Data Quality Management (ADQM) group for compliance with the laboratory SOP and usability according to a prepared checklist (see Appendix H). Draft results and the supporting raw data will not be deleted or discarded. Comments from review of the data package will be provided to the laboratory who will generate a revised laboratory data package, if necessary. An electronic data deliverable (EDD, see Appendix I) will also be provided by the laboratory and uploaded to the Locus EIM™ database.

All data will be reviewed using the Data Verification Module (DVM). The DVM is an internal review process used to assist with the determination of data usability. The electronic data deliverables received from the laboratory are loaded into the Locus EIM™ database and processed through a series of data quality checks, which are a combination of software (Locus EIM™ database Data Verification Module (DVM)) and manual reviewer evaluations. The data is evaluated against the following data usability checks:

- Field and laboratory blank contamination
- USEPA hold time criteria
- Missing Quality Control (QC) samples
- Matrix spike (MS)/matrix spike duplicate (MSD) recoveries and the relative percent differences (RPDs) between these spikes
- Laboratory control sample (LCS)/control sample duplicate (LCSD) recoveries and the RPD between these spikes
- Surrogate spike recoveries for organic analyses
- RPD between field duplicate sample pairs
- RPD between laboratory replicates for inorganic analyses
- Difference / percent difference between total and dissolved sample pairs, if any.

The DVM applies the following data evaluation qualifiers to analysis results, as warranted.

Qualifier	Definition
R	Unusable result. Analyte may or may not be present in the sample.
B	Not detected substantially above the level reported in the laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected. Reporting limit may not be accurate or precise.

The individual DVM narrative report for each lot entered into the EIM database will summarize which samples were qualified, the specific reasons for the qualification, and the potential bias in reported results.

The DVM review process described above will be performed on 100% of the data generated for the sampling event. The DVM review process will be supplemented by a manual review of the instrument-related QC results for calibration standards, blanks, and recoveries (Appendix H) to elevate the overall review process to be consistent with Stage 2b of the USEPA Guidance for Labelling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R-08-005, 2009).

Data Validation: Environmental Standards

Ten percent of the data points will be validated by a third-party reviewer, such as Environmental Standards, Inc., Valley Forge, Pennsylvania for compliance with the laboratory SOP and data usability, as appropriate. The *National Functional Guidelines* will be used as a guide for report formatting and application of qualifiers. Validation will take place concurrent with data reporting in order to expedite reporting of results. A formal report will be generated by the validator, which will include judgments on data usability and data qualifiers applied by the validator. The procedures that the Environmental Standards data reviewers will use to validate PFAS data for this project are described in the Data Validation SOP (Appendix J).

**QAPP Worksheet #37: Data Usability Assessment
(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)**

This worksheet documents procedures that will be used to perform the data usability assessment. The data usability assessment is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase, which involves a qualitative and quantitative evaluation of environmental data to determine if the project data are of the right type, quality, and quantity to support the decisions that need to be made. It involves a retrospective evaluation of the systematic planning process, and, like the systematic planning process, involves participation by key members of the project team. The data usability assessment evaluates whether underlying assumptions used during systematic planning are supported, sources of uncertainty have been accounted for and are acceptable, data are representative of the population of interest, and the results can be used as intended, with the acceptable level of confidence.

Identify personnel (organization and position/title) responsible for participating in the data usability assessment:

Chemours Project Director
AECOM Project Manager
AECOM Project Technical Adviser
AECOM Project QA Officer
AECOM Project Chemist
AECOM Field Task Leader

Describe how the usability assessment will be documented:

Summarize the data usability assessment process including statistics, equations, and computer algorithms that will be used to analyze the data:

Step 1	Review the project's objectives and sampling design <i>Review the key outputs defined during systematic planning (i.e., PQOs or DQOs and MPCs) to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.</i>
---------------	--

Step 2	Review the data verification and data validation outputs <i>Review available QA reports, including the data verification and data validation reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, non-compliant PT sample results, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.</i>
Step 3	Document data usability and draw conclusions <i>Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss data quality indicators. Assess the performance of the sampling design and identify limitations on data use. Update the conceptual site model and document conclusions. Prepare the data usability summary report that can be in the form of text and/or a table.</i>

Tables

Table 1
Laboratory QC Limits for Precision and Accuracy
PFAS Drinking Water Sampling Program QAPP

Analyte	CAS Num	Type	LCS/LCSD/MS/MSD Limits		
			Rec. Low %	Rec. High %	Precision %
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	958445-44-8	ADONA	70	130	30
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	756426-58-1	9CI-PF3ONS	70	130	30
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11CI-PF3OUdS	70	130	30
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	70	130	30
N-ethyl perfluorooctane sulfonamidoacetic acid	2991-50-6	NEtFOSAA	70	130	30
N-methyl perfluorooctane sulfonamidoacetic acid	2355-31-9	NMeFOSAA	70	130	30
Perfluorobutanesulfonic acid (PFBS)	375-73-5	PFBS	70	130	30
Perfluorodecanoic acid (PFDA)	335-76-2	PFDA	70	130	30
Perfluorododecanoic acid (PFDoA)	307-55-1	PFDoA	70	130	30
Perfluoroheptanoic acid (PFHpA)	375-85-9	PFHpA	70	130	30
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	PFHxS	70	130	30
Perfluorohexanoic acid (PFHxA)	307-24-4	PFHxA	70	130	30
Perfluorononanoic acid (PFNA)	375-95-1	PFNA	70	130	30
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	PFOS	70	130	30
Perfluorooctanoic acid (PFOA)	335-67-1	PFOA	70	130	30
Perfluorotetradecanoic acid (PFTeA)	376-06-7	PFTA	70	130	30
Perfluorotridecanoic Acid (PFTriA)	72629-94-8	PFTriDA	70	130	30
Perfluoroundecanoic acid (PFUnA)	2058-94-8	PFUnA	70	130	30

Table 1
Laboratory QC Limits for Precision and Accuracy
PFAS Drinking Water Sampling Program QAPP

Analyte	CAS Num	Type	Low Level LCS/LCSD/MS/MSD Limits		
			Rec. Low %	Rec. High %	Precision %
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	958445-44-8	ADONA	50	150	50
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	756426-58-1	9CI-PF3ONS	50	150	50
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11CI-PF3OUdS	50	150	50
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	50	150	50
N-ethyl perfluorooctane sulfonamidoacetic acid	2991-50-6	NEtFOSAA	50	150	50
N-methyl perfluorooctane sulfonamidoacetic acid	2355-31-9	NMeFOSAA	50	150	50
Perfluorobutanesulfonic acid (PFBS)	375-73-5	PFBS	50	150	50
Perfluorodecanoic acid (PFDA)	335-76-2	PFDA	50	150	50
Perfluorododecanoic acid (PFDoA)	307-55-1	PFDoA	50	150	50
Perfluoroheptanoic acid (PFHpA)	375-85-9	PFHpA	50	150	50
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	PFHxS	50	150	50
Perfluorohexanoic acid (PFHxA)	307-24-4	PFHxA	50	150	50
Perfluorononanoic acid (PFNA)	375-95-1	PFNA	50	150	50
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	PFOS	50	150	50
Perfluorooctanoic acid (PFOA)	335-67-1	PFOA	50	150	50
Perfluorotetradecanoic acid (PFTeA)	376-06-7	PFTA	50	150	50
Perfluorotridecanoic Acid (PFTriA)	72629-94-8	PFTriDA	50	150	50
Perfluoroundecanoic acid (PFUnA)	2058-94-8	PFUnA	50	150	50

Table 2
Laboratory Internal Standard and Surrogate Recovery Limits
PFAS Drinking Water Sampling Program QAPP

	Analyte	Rec. Low %	Rec. High %
Surrogate	13C2 PFDA	70	130
Surrogate	13C2 PFHxA	70	130
Internal Standard	13C2 PFOA	70	140
Internal Standard	13C4 PFOS	70	140
Internal Standard	d3-NMeFOSAA	70	140
Surrogate	d5-NEtFOSAA	70	130
Surrogate	13C3 HFPO-DA	70	130

Table 3
Laboratory Reporting Limits
PFAS Drinking Water Sampling Program QAPP

Analyte	Analyte Abbreviation	CAS Num	MDL ng/L	RL ng/L
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	ADONA	958445-44-8	0.5	2.0
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	0.5	2.0
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	0.5	2.0
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	0.5	2.0
N-ethyl perfluorooctane sulfonamidoacetic acid	NEtFOSAA	2991-50-6	0.5	2.0
N-methyl perfluorooctane sulfonamidoacetic acid	NMeFOSAA	2355-31-9	0.5	2.0
Perfluorobutanesulfonic acid (PFBS)	PFBS	375-73-5	0.5	2.0
Perfluorodecanoic acid (PFDA)	PFDA	335-76-2	0.5	2.0
Perfluorododecanoic acid (PFDoA)	PFDoA	307-55-1	0.5	2.0
Perfluoroheptanoic acid (PFHpA)	PFHpA	375-85-9	0.5	2.0
Perfluorohexanesulfonic acid (PFHxS)	PFHxS	355-46-4	0.5	2.0
Perfluorohexanoic acid (PFHxA)	PFHxA	307-24-4	0.5	2.0
Perfluorononanoic acid (PFNA)	PFNA	375-95-1	0.5	2.0
Perfluorooctanesulfonic acid (PFOS)	PFOS	1763-23-1	0.5	2.0
Perfluorooctanoic acid (PFOA)	PFOA	335-67-1	0.5	2.0
Perfluorotetradecanoic acid (PFTeA)	PFTA	376-06-7	0.5	2.0
Perfluorotridecanoic Acid (PFTrIA)	PFTrDA	72629-94-8	0.5	2.0
Perfluoroundecanoic acid (PFUnA)	PFUnA	2058-94-8	0.5	2.0

Figures

Figure 1

AUDIT CHECKLIST
PFAS Drinking Water Sampling Program QAPP

PROJECT: _____ PROJECT MANAGER: _____

SITE LOCATION: _____

AUDITOR: _____ DATE: _____

Question	Yes	No	Comment/Documentation
Field:			
1. Was an on-site safety officer appointed?			
2. Did site personnel receive a copy of the site-specific sampling and analytical plan in a timely manner to allow for sufficient review?			
3. Are copies available in the field during sampling?			
4. Was a briefing held off site, before any site work was begun, to acquaint personnel with sampling equipment, assign field responsibilities, and review safety procedures?			
5. Do field personnel have a field notebook?			
6. Are the site survey grid stakes present?			
7. Do the number and location of samples collected follow the procedures as specified in the site-specific sampling and analysis plan?			
8. Are samples labeled?			
9. Are samples being collected following the procedures?			
10. Was a chain-of-custody form filled out for all samples collected?			
11. Are samples preserved as specified?			
12. Are the number, frequency, and type of samples (including blanks and duplicates) collected as described in the sampling analysis plan?			
13. Are the number, frequency, and type of measurements and observations taken as specified in the site-specific sampling and analysis plan?			

**AUDIT CHECKLIST
(Continued)**

Question	Yes	No	Comment/Documentation
Field:			
14. Are operating procedures for field equipment available?			
15. Is a record maintained of the calibration of field equipment?			
16. Is field equipment being calibrated as required?			
17. Are geophysical cross sections correlated to geologic data?			
18. Is safety equipment being used by field personnel?			
19. Is emergency safety equipment available as required in the health and safety plan?			
20. Are well designations clearly labeled (i.e., well numbers)?			
21. Are caps on wells locked if not being used?			

Figure 2

**CORRECTIVE ACTION REQUEST
PFAS Drinking Water Sampling Program QAPP**

Number: _____ Date: _____

To: _____

You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) prevent it from reoccurring. Your written response is to be returned to the project quality assurance officer by _____.

Condition:

Reference Documents: _____

Recommended Corrective Actions: _____

Originator	Date	Approval	Date	Approval	Date
------------	------	----------	------	----------	------

Corrective Action

(A) Resolution: _____

(B1) Prevention: _____

(B2) Affected Documents: _____

Signature: _____ Date: _____

Q.A. Followup

Corrective Action Verified By : _____ Date: _____

Appendices

Appendix A

Analytical Method

**Title: Determination of Selected Per- and Polyfluorinated Alkyl
Substances in Drinking Water by Solid Phase Extraction (SPE) and
Analysis by Liquid Chromatography/Tandem Mass Spectrometry
(LC/MS/MS)
[Methods 537 and 537.1]**

Approvals (Signature/Date):			
	<u>04/30/2020</u>		<u>04/30/2020</u>
Robert Hrabak Technical Manager	Date	Joe Schairer Health & Safety Manager / Coordinator	Date
	<u>04/30/2020</u>		<u>04/30/2020</u>
Lisa Stafford Quality Assurance Manager	Date	Chris Williams Laboratory Manager	Date

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1 SCOPE AND APPLICATION

- 1.1 This procedure is based upon EPA Methods 537 Version 1.1 (September 2009) and 537.1 Version 1.0 (November 2018).
- 1.2 This method covers the determination of selected per- and polyfluorinated alkyl substances (PFAS) in drinking water using Liquid Chromatography with tandem Mass Spectrometry (LC/MS/MS). The specific compounds and their minimum reporting limits (RL) are indicated below.

TABLE 1.2 Analytes and Detection Limits			
Parameter		RL	
Analyte	CAS Num	Limits	Units
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	0.002	µg/L
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	919005-14-4	0.002	µg/L
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	0.002	µg/L
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	13252-13-6	0.002	µg/L
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	0.002	µg/L
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	0.002	µg/L
Perfluorobutanesulfonic acid (PFBS)	375-73-5	0.002	µg/L
Perfluorodecanoic acid (PFDA)	335-76-2	0.002	µg/L
Perfluorododecanoic acid (PFDoA)	307-55-1	0.002	µg/L
Perfluoroheptanoic acid (PFHpA)	375-85-9	0.002	µg/L
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	0.002	µg/L
Perfluorohexanoic acid (PFHxA)	307-24-4	0.002	µg/L
Perfluorononanoic acid (PFNA)	375-95-1	0.002	µg/L
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	0.002	µg/L
Perfluorooctanoic acid (PFOA)	335-67-1	0.002	µg/L
Perfluorotetradecanoic acid (PFTA)	376-06-7	0.002	µg/L
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	0.002	µg/L
Perfluoroundecanoic acid (PFUnA)	2058-94-8	0.002	µg/L

*Note: Abbreviation in parenthesis is the abbreviation listed in the reference method
 RL is equivalent to the MRL in the reference methods.*

(1) In some literature, the acronym ADONA refers to the ammonium salt, CAS 958445-44-8, and DONA refers to the parent acid. In Method 537.1, ADONA refers to the parent acid. DONA is the acronym present on the laboratory raw data.

- 1.3 When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.
- 1.4 When performing analysis on drinking water samples for compliance in Wisconsin, the requirements of work instruction WS-WI-0053, “Wisconsin DNR Requirements” must be reviewed and followed.

2 SUMMARY OF METHOD

- 2.1 A 250 mL aliquot of aqueous sample, preserved with dechlorinating agent, is loaded onto a 6-cc SPE cartridge containing 500 mg of polystyrenedivinylbenzene (SDVB) packing. The PFAS compounds are eluted with methanol and the methanol eluent is concentrated to dryness with nitrogen in a heated water bath, spiked with internal standard, and adjusted to 1.0 mL final volume with 96:4 methanol-water. Alternatively, for non-compliance samples, the extract can be adjusted to a 10 mL final volume with the same composition after elution.
- 2.2 A fixed volume, such as 10 µL, is injected on the HPLC equipped with a C₁₈ column interfaced to a tandem mass spectrometer (LC/MS/MS). The compounds are identified by comparing the acquisition of the mass transition and retention times to reference spectra and retention times for the calibration standards acquired under identical LC/MS/MS conditions.
- 2.3 The concentration of each analyte is determined by using internal standard technique. Surrogate analytes are added to all field and QC Samples to monitor the extraction efficiency of the method analytes and are quantified by internal standard technique.

3 DEFINITIONS

- 3.1 The laboratory employs nicknames and abbreviations for the PFAS compounds that do not always match those in the reference method or other literature. A key for those not present in Table 1.2 is below.

Table 3.1 Laboratory Nicknames		
Analyte	Laboratory Nicknames	CAS Num
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	F53B Minor	763051-92-9
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	DONA	919005-14-4
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	F53B Major	756426-58-1
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	GenX	13252-13-6
Perfluorotetradecanoic acid (PFTA)	PFTeA	376-06-7
Perfluorotridecanoic acid (PFTTrDA)	PFTriA	72629-94-8

- 3.2 Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).
- 3.3 Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottle and caps, glassware, solvent bottles, squirt bottles, and other processing apparatus. The analytes in this method can also be found in many common laboratory supplies and equipment, such as polytetrafluoroethylene (PTFE) products, LC solvent lines, methanol, aluminum foil, and SPE manifold and sample transfer lines. All items such as these must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks. Subtracting blank values from the sample results is not permitted.
- 4.2 Matrix interferences may also be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the sample. Humic and/or fulvic material is co-extracted by this method and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.
- 4.3 The potential exists for trace level organic contaminants in preservatives that are added to sample bottles. Trizma® is the chemical buffering and dechlorinating agent used for this method. Interference from these sources should be monitored by analysis of the laboratory reagent blanks, particularly when new lots of reagents are acquired.
- 4.4 SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide information regarding to the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that the contamination does not preclude analyte identification and quantitation.

5 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- 5.1.1 When briskly shaking the pH test strip and free-chlorine test strip after dipping, ensure that the test strip is only shaken away from you (into the hood). Do not shake the test strip back towards you.
- 5.1.2 Ensure that the vacuum exhaust hose used during the filtering is securely anchored inside of a fume hood so that vapors are not pumped into the working environment.
- 5.1.3 Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. PVC gloves provide adequate levels of protection against the chemicals used in this SOP.
- 5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.5 Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of SPE device and other laboratory equipment 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.

5.2 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
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.
Trizma® preset crystals, pH 7.0 (2-0-0)	Irritant	None listed	Inhalation, ingestion, eye or skin contact can cause irritation, coughing, shortness of breath, nausea, vomiting, redness and itching.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6 EQUIPMENT AND SUPPLIES

6.1 Auto sampler vials, 300 µL, polypropylene, with polypropylene screw caps or Agilent PTFE lined LC screw caps.

Note: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE lined septa. However, polypropylene caps do not reseal and evaporation occurs after injection. Multiple injections from the same vial is not recommended unless it is immediate and for qualitative purpose only.

6.2 Balance – Analytical capable of accurately weighing to 0.0001 g.

6.3 Bottles, 4 mL, 8 mL, and 250 mL size polypropylene, with polypropylene screw caps.

6.4 Extract concentrator or nitrogen manifold with water bath heating to 65°C.

6.5 Free-Chlorine Test Strips, designed to resist the interference from monochloramines and detection to 0.01 ppm, Fisher PN NC9116053, or equivalent

6.6 Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) –The instrumentation consists of an HPLC equipped with a refrigerated auto sampler, an injection valve, a column heater, and a pump capable of variable flow rate connected to a tandem MS/MS operated in negative ion electrospray (ESI) mode. Data is processed using Chrom Peak Review, version 2.3 or equivalent.

6.6.1 SCIEX 5500 Triple Quad MS. The Tandem MS/MS is operated in negative ion electrospray (ESI) mode.

6.6.2 Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

- 6.6.3 Phenomenex Gemini C₁₈ 3 μm, 2 mm x 50 mm, Part No. 00B-4439-B0, or equivalent.
- 6.6.4 PFC Isolator column, Phenomenex Luna C₁₈ 5 μm, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent.
- 6.6.5 Data acquired using Sciex Native, Version 1.6.2 or 1.6.3
- 6.6.6 Data is processed using Chrom Peak Review, Version 2.3 or higher.
- 6.6.7 Preventative and Routine Maintenance is described in the table below:

TABLE 6.6 HPLC/MS/MS Preventative Maintenance
<p><u>As Needed:</u> Change pump seals. Change in-line filters in auto sampler (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.</p>
<p><u>Daily (When in use)</u> 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.</p>
<p><u>Semi-Annually</u> Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.</p>
<p><u>Annually</u> Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.</p>

- 6.7 Narrow range pH test paper encompassing pH 6 to 8, Whatman part number 2629 990, or equivalent.

- 6.8 Pipettes, auto-pipets, and other equipment used to prepare standards and reagents.
- 6.9 Solid phase extraction (SPE) system.
 - 6.9.1 SPE Cartridges – 500 milligram/6-cc SPE cartridges containing polystyrenedivinylbenzene (SDVB) sorbent phase (Agilent PN 12255021), or equivalent.
 - 6.9.2 Vacuum extraction manifold – Supelco Visiprep, or equivalent. A manual vacuum manifold with column adapters, disposable liners, and column reservoirs for cartridge extraction.
- 6.10 Volumetric flask, Class A, various size 2.0 mL to 100 mL, as appropriate.
- 6.11 Test tubes, 15 mL, polypropylene, with polypropylene screw caps.
- 6.12 Vortex Mixer

7 REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

- 7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on 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 use without lessening the accuracy of the determination.
- 7.2 Ammonium Acetate, $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$, HPLC Grade, Fisher PN A639-500, or equivalent.
- 7.3 Ammonium Acetate/Reagent water, 20 mM – Prepared by weighing 1.54 grams of ammonium acetate and dissolving in 1 L of water. The resultant solution is prone to volatility and should be replaced every 48 hours or sooner.
- 7.4 Methanol, HPLC Grade, or equivalent.
- 7.5 Methanol-Water, 96:4 vol./vol., prepared by mixing 960 mL methanol and 40 mL reagent water. Stored in a polypropylene bottle and sealed with a screw cap.
- 7.6 Trizma® preset crystals, pH 7.0 (Sigma Aldrich Cat. No. T7193-250G or equivalent), a mixed blend of Tris [Tris(hydroxymethyl) aminomethane] and Tris HCl [Tris(hydroxymethyl)aminomethane hydrochloride] which function as a buffer and removes chlorine in chlorinated finished water. It is added to the sample bottle at a concentration of 5 g/L (or 1.25 g per 250 mL polypropylene bottle). Bottleneckware with the appropriate amount Trizma® already present may be purchased and used.

- 7.7 Reagent Water - HPLC grade water, distilled or de-ionized water, free of chemicals of interest , i.e. <1/3 RL.
- 7.8 Standards
- Prior to use, allow all solutions to warm to room temperature. Mix using a vortex mixer prior to taking aliquots for use.*
- 7.8.1 Individual and mixed stock solutions are available from reputable vendors such as Wellington Laboratories. However, the source standards for PFOS and PFHxS, according to Methods 537 and 537.1, must contain both the linear and branched isomers. These standards must be purchased from other sources such as Santa Cruz Biotechnology and Sigma-Aldrich, when needed.
- 7.8.2 Manufacturer will usually have an expiration date on the standards. If not, a two year from receipt of the standards is used for native compounds and five years for labeled or deuterated compounds.
- 7.8.3 Individual stock solutions purchased from vendors such as Wellington Laboratories are typically at 50 µg/mL in methanol. These are referred to as the 537 Stock Solutions
- 7.8.4 A mixture of PFAS from Absolute Standards or Wellington Laboratories is used as a second source and the concentration is at 2 µg/mL, however, if branched and linear isomers of PFC standards are available, they should be purchased and used for this method. Second source standards are purchased from different vendors when possible or different lots if a different vendor is not available.
- 7.8.5 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
- 7.8.6 **537 Working Standard** – An appropriate amount (see table below) of the 537 Stock solutions (Section 7.8.3) are added to a 10 mL volumetric flask with 400 µL of water and diluted to mark with methanol. The resultant mixture of the 537 Working Standard is 1.0 µg/mL 96% methanol / 4% water and is stored in a polypropylene bottle at 0 – 6°C. The solution is valid for 6 months. This solution is equivalent to the primary dilution standard in the reference methods.
- 7.8.7 **537 IM Standard** – Dilute 1.0 mL of the 537 Working Standard to a 10 mL volumetric flask, add 360 µL of water and dilute to mark with methanol. The resultant mixture of the 537 IM standard is 0.1 µg/mL 96%

methanol / 4% water and is stored in a polypropylene bottle at 0 – 6°C. The solution is valid for 6 months.

TABLE 7.8.7 Composition of Working and IM Standard Solutions				
Analyte	537 Stock Conc. (µg/mL)	Aliquot (mL)	537 Working Standard Conc. (µg/mL)	537 IM Standard Final Conc. (µg/mL)
PFBS ⁽¹⁾	50	0.2	1.0	0.1
PFHxS ⁽¹⁾	50	0.2	1.0	0.1
PFOA	50	0.2	1.0	0.1
PFOS ⁽¹⁾	50	0.2	1.0	0.1
PFHpA	50	0.2	1.0	0.1
PFNA	50	0.2	1.0	0.1
PFDA	50	0.2	1.0	0.1
PFDoA	50	0.2	1.0	0.1
PFHxA	50	0.2	1.0	0.1
PFTeA	50	0.2	1.0	0.1
PFTriA	50	0.2	1.0	0.1
PFUnA	50	0.2	1.0	0.1
NEtFOSAA	50	0.2	1.0	0.1
NMeFOSAA	50	0.2	1.0	0.1
HFPO-DA	50	0.2	1.0	0.1
F53B Minor	50	0.2	1.0	0.1
F53B Major	50	0.2	1.0	0.1
ADONA ⁽²⁾	52	0.2	1.0	0.1
DONA ⁽²⁾	50	0.2	1.0	0.1

Note (1): The concentration listed is based on the salt. When entered into the standards tracking software and subsequently used for quantitation, the value is corrected to the anion.

Note (2): DONA is the laboratory abbreviation for 4,8-dioxa-3H-perfluorononanoic acid, CAS 958445-44-8. “ADONA” is used in the laboratory to refer to the ammonium salt, which is calculated by multiplying the DONA concentration by 1.048.

7.8.8 **537 Spike Mixes** – The high-level and mid-level spikes are made by adding each of the 50 µg/mL individual stock solutions to 96% methanol / 4% water as listed below:

- 537-HSP 100 µg/L 500 µL of each solution to 250 mL
- 537-MSP 50 µg/L 250 µL of each solution to 250 mL

Dilute the 537-HSP solution in 96% methanol / 4% water as indicated below to make the 537 low-level spike:

- 537-LSP 2 µg/L 5.0 mL of 537-HSP to 250 mL

- 7.8.9 **537 IM Surrogate solution** – Prepare a 537 IM Surrogate Mix by adding appropriate amount of the stock solution to a 10.0 mL volumetric flask, add 400 uL of water and dilute to mark with methanol. The resultant mixture is 0.1 µg/mL 96% methanol / 4% water. The solution is stored in a polypropylene bottle at 0 – 6°C and is valid for 6 months.
- 7.8.10 **537 Surrogate Mix** – Dilute 0.2 mL of the 50 µg/mL stock solution, with 8 mL of water and dilute to a final volume of 200 mL in methanol for a 1000x dilution. The resultant mixture is 0.05 µg/mL 96% methanol / 4% water. The solution is stored in a polypropylene bottle at 0 – 6°C and is valid for 6 months.

TABLE 7.8.10 Composition of 537 Surrogate Mix			
537 Surrogate Analyte	Stock Conc. (µg/mL)	Aliquot (mL) to 10mL final volume	537 Surr Mix Conc. (µg/mL)
13C2-PFHxA	50	0.20	0.10
13C2-PFDA	50	0.20	0.10
d5-NEtFOSAA	50	0.20	0.10
13C3-HFPO-DA	50	0.20	0.10

- 7.8.11 **537 IS solution** – A 537 Internal Standard Mix is prepared by diluting the appropriate amount of stock solution with 8 ml of water to a 200 mL volumetric flask and dilute to mark with methanol. The resultant mixture is 0.05 µg/ml 96% methanol / 4% water. , stored in a polypropylene bottle at 0 – 6°C, and is valid for 6 months.

TABLE 7.8.11 Composition of 537 IS Mix			
537 Internal Standard Analyte	Stock Conc, (mg/mL)	Aliquot (mL) to 10mL final volume	537 IS Solution Conc. (ug/mL)
13C2-PFOA	50	0.2	0.05
13C4-PFOS ⁽¹⁾	50	0.2	0.05
d3-NMeFOSAA	50	0.2	0.05

Note (1): The standard is received as salt form, the concentration is listed as anion form.

- 7.8.12 **537 Calibration curve** – Refer to the table below for the preparation of the calibration curve. Dilute each level with 96% methanol / 4% water. The calibration curve solutions are stored in polypropylene bottles at 0 – 6°C

and are valid for 6 months.

TABLE 7.8.12A Recipes for 537 Calibration Solutions							
537 Standards	Volume (µL) to add in 20 mL FV						
	L1	L2	L3	L4	L5	L6	L7
537 High Spike (0.1 µg/mL)	50	100	500	X	X	X	X
537 Working Standard (1.0 µg/mL)	X	X	X	200	500	1000	2000
537 Surrogate Mix (0.50 µg/mL)	10,000	10,000	10,000	10,000	10,000	10,000	10,000
537 IS Mix (0.05 µg/mL)	10,000	10,000	10,000	10,000	10,000	10,000	10,000

TABLE 7.8.12B Composition of Calibration Solutions								
Analyte		Standard Level - Concentration as ng/mL						
		L1	L2	L3	L4	L5	L6	L7
PFBS		0.25	0.5	2.5	10	25	50	100
PFHxS		0.25	0.5	2.5	10	25	50	100
PFOA		0.25	0.5	2.5	10	25	50	100
PFOS		0.25	0.5	2.5	10	25	50	100
PFHpA		0.25	0.5	2.5	10	25	50	100
PFNA		0.25	0.5	2.5	10	25	50	100
PFDA		0.25	0.5	2.5	10	25	50	100
PFDaA		0.25	0.5	2.5	10	25	50	100
PFHxA		0.25	0.5	2.5	10	25	50	100
PFTeA		0.25	0.5	2.5	10	25	50	100
PFTriA		0.25	0.5	2.5	10	25	50	100
PFUnA		0.25	0.5	2.5	10	25	50	100
NEtFOSAA		0.25	0.5	2.5	10	25	50	100
NMeFOSAA		0.25	0.5	2.5	10	25	50	100
HFPO-DA		0.25	0.5	2.5	10	25	50	100
F53B Minor		0.25	0.5	2.5	10	25	50	100
F53B Major		0.25	0.5	2.5	10	25	50	100
DONA		0.25	0.5	2.5	10	25	50	100
SU	13C2-PFHxA	25	25	25	25	25	25	25
SU	13C2-PFDA	25	25	25	25	25	25	25
SU	d5-NEtFOSAA	25	25	25	25	25	25	25
SU	13C3-HFPO-DA	25	25	25	25	25	25	25
IS	13C2-PFOA	25	25	25	25	25	25	25
IS	13C4-PFOS	25	25	25	25	25	25	25
IS	d3-NMeFOSAA	25	25	25	25	25	25	25

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 unconcentrated (non-compliance sample) extracts is 1/10th the levels indicated above.

- 7.8.13 Second source standard is either from a separate vendor or a separate lot number from the primary stock standards. The standard is stored at 0 – 6°C. Unless specified by the manufacturer, an expiration date of six months from receipt of the standards is used.
- 7.8.14 Second source standard is not needed for the surrogate and IS compounds.
- 7.8.15 **Second Source (ICV) Stock Solution** –Stock solutions prepared from neat are diluted in methanol at 2.0 µg/mL. They are stored in a polypropylene bottle at 0 – 6°C and the solution is valid for six months. Alternatively, a mixed stock solution may be used, for example part number 99896 from Absolute Standards).

Level	Final Volume	537 ICV Stock (2µg/mL)	537 IM Surrogate Mix (0.1µg/mL)	537 IS Mix (0.1µg/mL)
ICV	10 mL	100 µL	5.0 mL	5.0 mL

- 7.8.16 **537 ICV Standard Mix** – is prepared by diluting the second source 2.0 µg/mL (PFAC-24PAR) ICV stock solution into a 200 mL volumetric flask and dilute to mark with the appropriate composition of 96% methanol / 4% water. 96:4 in 200 mL is 192 mL of methanol and 8 mL of water. The resultant mixture is 2 µg/L, stored in a polypropylene bottle at 0 – 6°C, and is valid for 6 months.

Analyte	ICV Concentration (ng/mL)
PFBS	20
PFHxS	20
PFOA	20
PFOS	20
PFHpA	20
PFNA	20
PFDA	20
PFDoA	20
PFHxA	20
PFTeA	20
PFTriA	20

Analyte	ICV Concentration (ng/mL)	
PfUnA	20	
NEtFOSAA	20	
NMeFOSAA	20	
HFPO-DA	20	
F53B Minor	20	
F53B Major	20	
DONA	20	
SU	13C2-PFHxA	25
SU	13C2-PFDA	25
SU	d5-NEtFOSAA	25
SU	13C3-HFPO-DA	25
IS	13C2-PFOA	25
IS	13C4-PFOS	25
IS	d3-NMeFOSAA	25

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE FOR PFC

- 8.1 A 250 mL polypropylene bottle with a polypropylene screw cap is recommended for sample collection. High-density polyethylene (HDPE) containers with HDPE screw caps may also be used, based on the availability of the containers. Prior to shipment to the field, the buffering and dechlorinating reagent Trizma® (pH 7) is added as a dry solid to each bottle so that the final amount is 5 g/L. For 250 mL size bottle, 1.25 g of Trizma® is added.
- 8.2 The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile glove will aid in minimizing this type of accidental contamination of the samples.
- 8.3 Sample bottles must not be pre-rinsed with sample before collection.
- 8.4 Fill sample bottles, taking care not to flush out the sample preservative. Samples do not need to be collected headspace free.
- 8.5 After collecting the sample cap carefully to avoid spillage, and agitate by inverting a few times. Keep samples sealed until analysis.
- 8.6 Field reagent blanks (FRB) must accompany with the sample set. The sample set is composed of samples collected from the sample site and at the same time.
 - 8.6.1 At the laboratory, a sample bottle is filled with reagent water and preservatives, sealed, and shipped to the sampling site along with the

sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) is also shipped.

- 8.6.2 At the sampling site, the sampler must open the shipped FRB and pour the reserved reagent water into the empty shipped bottle, seal and label this bottle as the FRB.
- 8.6.3 The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFASs were not introduced into the sample during sample collection and handling.
- 8.6.4 The preservative used for the FRB must be from the same lot number as is used for the samples.
- 8.7 Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours following collection. Sample temperature must be confirmed to be at or below 10°C when samples are received at the laboratory.
- 8.8 A free chlorine test must also be performed when samples are received at the laboratory to verify that free chlorine has been neutralized. If free chlorine is present, then the sample is not acceptable for this method – the client must be notified for possible re-sampling. File an NCM for inclusion in the final report in TALS, and ensure that it is emailed to the project manager.
- 8.9 Samples stored in the laboratory must be stored below 6°C until extraction, but it should not be frozen. Do not freeze.
- 8.10 Aqueous samples must be extracted within 14 days of sampling when samples are properly preserved, shipped, and stored.
- 8.11 Extracts must be stored at room temperature and analyzed within 28 days after extraction.

Note: For drinking water samples, nonconformances for items 8.9 through 8.11 will result in non-compliant results that will be rejected by the regulator.

9 QUALITY CONTROL

- 9.1 Initial Demonstration of Capability
 - 9.1.1 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, and it is mandatory to analyze prepared extracts on the same instrument.

- 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 required by the program or client. In the event that multiple MS/MSD are run with a batch due to client requirements, the additional MS/MSD 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. The method blank is processed in the same manner, including the addition of dechlorinating agent, anti-microbial agents, and pH adjustments on, and at the same time, as the associated samples. Corrective actions must be documented on a Non-Conformance memo and implemented when target analytes are detected in the method blank above 1/3 the reporting limit or when surrogate 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.
- 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 Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the reporting limit, i.e. $MB < 1/3 RL$.
- 9.3.3 Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- 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 fortification level must be rotated between low-, mid- and high-level spikes from batch to batch.
- 9.4.1 The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner (including the addition of dechlorinating and anti-microbial chemicals) 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.

- 9.4.2 The low concentration LCS must be no greater than 2x MRL.
- 9.4.3 Similarly, the high concentration LCS should be near the high end of the calibration range established during the initial calibration.
- 9.4.4 The percent recovery (%R) for each analyte is calculated using the equation:

$$\%R = (A-B)/C \cdot 100$$

Where:

A = measured concentration in the fortified sample.

B = measured concentration in the unfortified sample

C = expected fortified concentration.

- 9.4.5 For samples fortified at mid or high levels, the acceptance limits are 70-130%. For samples fortified at the low level at or near the RL (within 2x RL) the acceptance limits are 50 to 150%.
- 9.4.6 If the analyte(s) recovery do not meet the criteria and the CCV standard is in control, a second injection should be done to determine if there was a bad injection.
- 9.4.7 If the analyte(s) is in control, report the valid injection.
- 9.4.8 If the analyte(s) is out of control, the data for this problem analyte(s) must be considered invalid for all samples in the extraction batch.
- 9.5 Surrogate recoveries for the Method Blank, LCS, and samples must be within the limits of 70% to 130%.

- 9.5.1 Surrogate recovery is calculated using the following equation:

$$\% R = (A/B) \cdot 100$$

Where

A = calculated SURR concentration for the QC or field sample

B = Fortified concentration of the SURR.

- 9.5.2 If the surrogate recovery is less than 70% or greater than 130%, check the calculation to locate possible errors, check for standard degradation, contamination, and instrument performance. Correct the problem and re-analyze the extract.
- 9.5.3 If the extracts reanalysis fails the 70-130% criterion, the analyst should check the calibration by injecting and evaluating the last CCV that passed.

- 9.5.4 If the CCV standard fails for the surrogate compounds then recalibration and reinjection is in order.
- 9.5.5 If the CCV standard passes, then extraction of the sample should be repeated provided the sample is still within the holding time. If the sample is past the holding time, the client must be immediately notified, as compliance samples typically require resampling.
- 9.5.6 If the re-extraction also fails the recovery criterion, immediately notify the client. Also file an NCM in TALS discussing the failure.
- 9.5.7 Alternatively, the client may elect to collect a new sample and re-analyze.
- 9.6 Internal Standard (IS) - must be monitored during each analysis day. Internal Standards is added to all blanks, standards and samples before analysis.
- 9.6.1 The IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration and must not deviate by more than 70-140% from the most recent CCV standard.
- 9.6.1.1 This is performed by assigning all non-CCV samples to be compared to both the last CCV and last ICIS sample within the Chrom method. (Method Editor, Limit Group, Edit, ISTD/Tune/Tail tab).
- 9.6.2 If the IS areas do not meet the criteria for samples, re-analyze the affected sample in a new capped autosampler vial. Random evaporation loss has been observed with PP caps causing high IS areas.
- 9.6.3 A poor injection, as well as matrix enhancement or suppression could cause the IS area to exceed these criteria. A second injection should be done to determine if there was a bad injection.
- 9.6.4 If the re-injected aliquot produces an acceptable IS response, report results for that aliquot.
- 9.6.5 If the re-injected aliquot failed again, the analyst should check the calibration by reanalyzing the most recently acceptable CCV standard.
- 9.6.6 If the CCV standard fails, recalibration is in order.
- 9.6.7 If the CCV standard is acceptable, extraction of the sample may need to be repeated provided the sample is still within the holding time. If the sample is outside the holding time, the client must be immediately notified, as compliance samples may be considered invalid without passing internal standards. If not re-extracted within the holding time, report the reinjected extract and file an NCM regarding the failed internal standards.

- 9.6.8 Alternatively, the client may elect to collect a new sample and re-analyze.
- 9.7 Continuing Calibration Verification (CCV) – is analyzed at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is valid.
- 9.7.1 The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required.
- 9.7.2 The CCV should vary throughout the run. A low level CCV is analyzed first, then subsequent CCV alternate between the mid and high level.
- 9.7.3 At the beginning of a run and if a curve has not been run that day, two levels of CCV should be run. One CCV should be run at the reporting limit and the other CCV at the mid point of the curve.
- 9.7.4 The reporting limit CCV should be within 50% of the true value and the mid and high level CCV should be within 30% of the true value.
- 9.8 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.
- 9.8.1 The MS/MSD fortification level must be rotated through low-, mid-, and high-level from batch to batch.
- The low concentration MS/MSD must be no greater than 2x MRL.
- 9.8.2 Analyte recoveries may exhibit matrix bias. Spiked analytes with recoveries or precision outside of the control limits may be reported if result for the LCS is in control, however, the results are considered suspect due to matrix effects and must be narrated as such.
- 9.8.3 For samples fortified at mid or high levels, the acceptance limits are 70-130% recovery and 30% RPD. For low level at or near the RL (within 2x RL) the acceptance limits are 50 to 150% recovery and 50% RPD. Corrective actions must be documented on a nonconformance memo and implemented when recoveries of any spiked analyte are outside of the control limits.
- 9.8.4 A duplicate control sample (LCSD or DCS) may be substituted when insufficient sample volume is provided to process an MS/MSD pair if required by the program or client. The LCSD is evaluated in the same manner as the LCS.

9.9 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. The ICV must meet mid-level CCV acceptance criteria, as detailed in Table 10.9.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

9.10 Field reagent blank (FRB) – A FRB should be submitted per sample set and extracted with the associated samples. While the analysis of the FRB is required only if there are detections in the associated client samples, it is generally analyzed at the same time as the client samples.

9.10.1 The FRB should not contain any analyte greater than 1/3 of the reporting limit.

9.10.2 If the associated samples contain any analytes at or above the reporting limit, evaluate the FRB. If the FRB has any detections greater than 1/3 of the reporting limit, contact the client for further guidance. Per the reference methods, all samples are considered invalid and required recollection and reanalysis.

9.10.3 For samples submitted for the state of Arizona, if a FRB is not present the data shall be qualified with the Arizona specific qualifier of T6, “Data cannot be used for compliance purposes.”, and file an NCM indicating such.

9.11 A typical run sequence for 537 would be as follows:

1. Primer (A number of primers until the instrument is stable)
2. Blank
3. Calibration Curve (A minimum of 5 calibration levels)
4. Blank + IS
5. CCV low-level (at MRL)
6. ICV
7. PFOA RT marker
8. MB
9. LCS
10. Sample 1
11. Sample 1 MS
12. Sample 1 MSD
13. Sample 2
14. etc. (up to 10 field samples between CCVs).
15. CCV mid or high-level (Rotate mid, and high level CCV throughout the sequence).

10 CALIBRATION

- 10.1 Mass tuning is performed by the service engineer following major maintenance to the mass spectrometer.
- 10.2 Initial Tune:
- 10.2.1 Mass calibration is verified on an as-needed basis, when conditions have been optimized, by acquiring a full-scan mass spectrum of the target analytes ions. The target analyte ions should be within 0.3 m/z of the expected mass.
- 10.2.2 Optimize the $[M-H]^-$ for each target analyte by infusing approximately 1.0 µg/mL of each analyte into a tee fitting with eluent flowing at about 0.1 mL/min. Set the entrance and exit slits to about 45, the collision to 0 and the collision gas off. Working with the parent ions for each analyte, vary the MS parameters (capillary voltage, temperatures, gas flows, etc.) until optimal analyte responses are determined. The target analytes may have different optimal conditions requiring some compromise on some of the conditions for the precursor ions.
- 10.2.3 With the infusion still running, next turn on the collision gas, set the entrance to about -5, the exit to about 1.5, and vary the collision voltage and collision gas to get optimal conditions for each analyte. A compromise setting for the collision gas will have to be made for product ions.
- 10.3 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.
- 10.4 With the exception of the circumstances delineated in policy CA-Q-P-003, "Calibration Curves and Selection of Calibration Points", 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.
- 10.5 The following requirements must be met for the initial calibration to be used.
- Response must increase with increasing concentration.
 - The internal standard area count must be within criteria for the standards.
 - The origin must be forced through zero (specified by methods 537 and 537.1).
 - There must be no carryover at or above 1/3 MRL after a high calibration standard.

- At least five calibration levels are used to prepare the initial calibration curve, however, for quadratic fit at least six calibration levels are used.
- 10.5.1 The Chrom data system is programmed to complement the calibration evaluation guidelines in policy CA-Q-P-003 by evaluating calibration curve fits in the order listed below. An optimal fit is recommended to the analyst, who may override based on evaluation of the residuals for each calibration level, as per policy CA-Q-P-003.
- Linear, 1/concentration² weighting, forced through zero
 - Linear, 1/concentration weighting, forced through zero
 - Linear, no weighting, forced through zero
 - Average Response Factor
 - Quadratic, no weighting, forced through zero
- 10.6 A fixed injection volume is used for quantitation purposes and is to be the same for samples and standards in the analytical sequence.
- 10.7 All units used in the calculations must be consistently uniform, such as concentration in ng/mL and the use of peak area.
- 10.8 Initial Calibration - At least five calibration levels are used to prepare the initial calibration curve, except for quadratic fit at least six calibration levels are used. Larger concentration range will require more calibration points. Each standard is injected once to obtain the peak area for each analyte at each concentration and the internal standard area count must be within criteria from each standard.
- 10.8.1 The lowest concentration of the calibration standard must be at or below the MRL, which may depend on system sensitivity. It is recommended that at least four of the calibration standards are at a concentration greater than or equal to the MRL.
- 10.8.2 The quantitation is performed using the Internal Standard technique. The data system software is used to generate the best fit; average response factor, a linear regression, or a quadratic fit for each of the analytes. The linear regression or quadratic fit **must always** be forced through zero and may be concentration weighted, if necessary. Forcing through zero allows for a better estimate of the background levels of the method analytes.
- 10.8.3 Peak Asymmetry Factor – a measure of peak tailing. The peak asymmetry factor must be calculated for the first two eluting peaks (if only two analytes are being analyzed, both must be evaluated) on a mid-level calibration standard.
- 10.8.4 The peak asymmetry factors must fall in the range of 0.8 to 1.5 using the equation below when the initial calibration curve is generated and during

the initial demonstration is performed.

Note: Modifying the standard or extract composition to more aqueous content to prevent poor peak shape is not permitted.

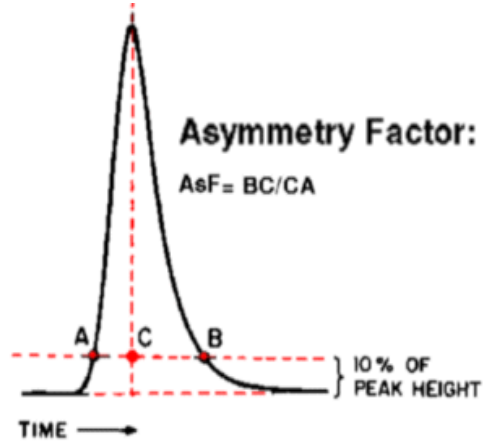


Figure 1 ^{ref. 16.4.6}

Where:

AsF = peak asymmetry factor

BC = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex.

AC = width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the peak apex. Attach this document to the calibration standard used for evaluation by scanning the document and associating it to the file as a document type of High Res MS Tune in TALS.

10.8.5 If peak asymmetry factor is not met or if peak broad, split or fronting peaks are observed for the first two eluting chromatographic peaks, change the initial mobile phase to higher aqueous content until the peak symmetry factor is met (AsF = 0.8 to 1.5).

10.8.6 Calculate the measured concentration for each analyte and surrogate in each calibration standard using the factors from the calibration curve. Compare the measured values to the true values. Each result must meet the criteria below:

Table 10.8 ICAL Criteria			
Standard Concentration	Criteria for Native Analyte	Criteria for Surrogate	Criteria for IS

Lowest calibration standard ≤ MRL	± 50% of true value	± 30% of true value	20% RPD
> MRL	± 30% of true value	± 30% of true value	20% RPD

- The isotopically labeled IS(s) in this method may undergo suppression in the ESI source if the concentration of the co-eluting unlabeled method analyte(s) is too high. The analyte concentration at which suppression may occur can vary depending on the instrument, LC conditions, ESI conditions, IS concentration, etc. To evaluate whether suppression is occurring during calibration, calculate the relative percent difference (RPD) between the high (H) and low (L) areas for each IS using the equation:

$$RPD = \frac{(H - L)}{(H + L)/2} \times 100$$

- The RPD calculated above must be <20% for each IS during calibration. If the calculated RPD is >20% for any IS, the analyst must recalibrate at lower analyte concentrations until the IS RPDs are <20%.
- Use the IS summary form (F7) from Chrom for this evaluation. Calculate the on this form. The form is then to be attached to the ICAL similarly to the asymmetry check.
- If these criteria cannot be met, it is recommended that corrective action is taken to reanalyze the calibration standards, restrict the range of calibrations, or select an alternate method of calibration (however, forcing the curve through zero is still required).
- Methods 537 and 537.1 specify five calibration concentrations of the initial calibration curve spanning a 20-fold concentration range. Standard preparation consists of 7 calibration levels; however, higher concentration levels are not required and may not meet the ICAL criteria.

10.9 Continuing calibration verification standard (CCV) – is injected prior to sample analysis (with the exception of after a curve and ICV a CCV is not needed until 10 field samples have been run), following every 10 field samples or fewer, and at the end of the analytical run. *See section 9.11 for a typical run sequence.*

10.9.1 The method blank (MB), laboratory control sample (LCS), and matrix spike/spike duplicate (MS/SD) are not counted as field samples.

10.9.2 The first CCV injected must be the low level standard at or below the MRL to verify instrument sensitivity prior to any analysis. If standards have been prepared such that all low calibration points are not in the same calibration solution, it may be necessary to analyze two calibration standards to meet this requirement.

- 10.9.3 Alternate subsequent CCV between the mid-level and high-level standards. If the initial calibration curve was not performed on the day of sample analysis, two CCV are required prior to analysis, the first one at or below the MRL, and the second one near the mid-level of the calibration curve.

Table 10.9 CCV Criteria			
Standard Concentration	Criteria for Native Analyte	Criteria for Surrogate	Criteria for IS
First CCV of batch (\leq MRL)	$\pm 50\%$ of true value	$\pm 30\%$ of true value	$\pm 50\%$ from average ICAL response & 70-140% from most recent CCV.
First CCV of batch ($>$ MRL)	$\pm 30\%$ of true value	$\pm 30\%$ of true value	$\pm 50\%$ from average ICAL response & 70-140% from most recent CCV.
Subsequent CCVs ($>$ MRL)	$\pm 30\%$ of true value	$\pm 30\%$ of true value	$\pm 50\%$ from average ICAL response & 70-140% from most recent CCV.

- 10.9.4 If the CCV standard is out of control, another injection of the standard may be injected singly to determine if the system is back in control. If it is still out of control, then there may be a problem with the standards, the instrument, or the column. If the column is replaced, then a new curve is necessary.

11 PROCEDURE

- 11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, and chemical characteristics, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Non-Conformance Memo shall be filed in the project file.
- 11.2 Any unauthorized deviation from this procedure must also be documented as a nonconformance, with a cause and corrective action described. See WS-QA-0023 for additional information on the established procedures for the identification and documentation of non-conformances and corrective actions.
- 11.3 Samples must be checked for verification of temperature when samples are received at the laboratory. As soon as possible after samples have arrived, free chlorine checks and pH checks should be done as well.
- 11.3.1 Check each sample for pH and presence of free chlorine. Record the

observations.

- 11.3.2 **For pH determination**, dip a pH test strip briefly into an aliquot of the sample in a disposable container.
- 11.3.3 Remove and shake the test strip once briskly to remove the excess sample.
WARNING: Ensure that the pH and free-chlorine test strips are shaken away from you (into the hood) and not toward you.
- 11.3.4 Compare the test strip to the reference chart to determine the pH of the sample.
- 11.3.5 Note the pH on the bench-sheet. If the value is different by 0.5 pH unit from 7.0, the sample was not sufficiently preserved. The Project Manager must be notified and a non-conformance memo must be filed.
- 11.3.6 If the pH was not within the range, either Trizma® was not used as a preservative in the sample or the sampling was not performed correctly. Continue with the free chlorine determination on the samples.
- 11.3.7 **For free chlorine determination**, dip a free-chlorine test strip into the aliquot and move it with a constant, gentle back and forth motion for 20 seconds.
- 11.3.8 Remove and shake the test strip once briskly to remove the excess sample.
WARNING: Ensure that the pH and free-chlorine test strips are shaken away from you (into the hood) and not toward you.
- 11.3.9 Wait 20 seconds then compare the test strip to the reference chart to determine the concentration (ppm or mg/L) of free chlorine. Complete the comparison within one minute.
- 11.3.10 Note and record the concentration of the free chlorine on the bench-sheet. If the value is greater than or equal to 0.1 mg/L, the sample is not sufficiently dechlorinated, and the sample is not acceptable for this method. The Project Manager must be notified immediately and a non-conformance memo must be filed.
- 11.4 Create MB and LCS samples by filling empty pre-preserved sample bottles with deionized water. Use bottles with the same lot number of Trizma as the client samples. In TALS, document this by entering “in all QC, field samples, and FRB” after the lot number in the Trizma ID field.
- 11.5 Sample volume may be determined by volume or by weight.
 - 11.5.1 To determine the sample volume by weight, weigh the field sample containers to the nearest 0.1g and record the value in the TALS batch as the
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“gross weight”. When the container is empty, weigh again and record the value as the “tare weight”. Assuming a density of 1.0 g/mL, the sample volume is calculated within the limits using the difference of the two weights.

11.5.2 To determine the sample volume by volume, use an indirect technique: mark the meniscus on the sample bottle prior to sample preparation. Once the bottle is empty, fill to the mark with tap water, and pour the tap water into a graduated cylinder to determine the volume to the nearest mL. Do not transfer sample to graduated cylinders for volume determination as PFASs may absorb to the surface.

11.6 Add the 537-Surrogate to all samples, including the MB, LCS, and MS/SD.

Note: Surrogate solution should be at room temperature and adequately vortexed to ensure contents are mixed well before using.

- Add 500 µL of the 0.1 µg/mL 537-Surr mix to a 250 mL sample for a final concentration of 40 ng/L.

11.7 One laboratory control sample (LCS) must be prepared with every batch – the LCS is rotated between low, mid, and high levels between subsequent batches.

Note: Spike solution should be at room temperature and adequately vortexed to ensure contents are mixed well before using.

Table 11.7 LCS Fortification				
Amount Added		Standard Concentration		Sample Concentration
Low	500 µL	537-LSP SPIKE	1.0 ng/mL	2 ng/L
Mid	500 µL	537-MSP SPIKE	10 ng/mL	20 ng/L
High	500 µL	537-HSP SPIKE	100 ng/mL	200 ng/L

11.8 The MS/MSD is rotated in the same manner as the LCS.

Table 11.8 MS/SD Fortification				
Amount Added		Standard Concentration		Sample Concentration
Low	500 µL	537-LSP SPIKE	1.0 ng/mL	2 ng/L
Mid	500 µL	537-MSP SPIKE	10 ng/mL	20 ng/L
High	500 µL	537-HSP SPIKE	100 ng/mL	200 ng/L

11.9 After the addition of the surrogate solution and spike solution, seal the sample bottles with screw caps and mix the contents well by inverting and shaking prior to proceeding.

11.10 Solid Phase Extraction (SPE)

Note: Avoid the use of Teflon products for this method, especially with the Teflon squirt bottles. The polyethylene (PE) bottle has been tested and found to be acceptable for the PFAS analysis. Cartridge conditioning and loading – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. If they do go dry, then the conditioning step must be started over.

- 11.10.1 Set up SPE cartridge for conditioning - 500 milligram/6-cc SDVB sorbent phase (Agilent PN 12255021) was used for the method validation of 537 and 537.1.

Warning: Ensure that the vacuum exhaust hose used during the filtering is securely anchored inside of a fume hood so that vapors are not pumped into the working environment.

- 11.10.2 Document the manufacturer and lot number of the SPE cartridge.

11.10.2.1 New SPE lots should be QC checked for PFAS recovery and background before using.

11.10.2.2 New source for the sorbent phase must undergo the IDOC, verify that all Quality Control (QC) acceptance criteria are met, and that acceptable method performance can be verified in a real sample matrix.

- 11.10.3 Condition the cartridges with 15 mL of methanol followed by 18 mL of water with vacuum. Stop the flow when the water reaches the top of the packing. If it goes dry, repeat the conditioning.

- 11.10.4 Add ~5 mL of water to each cartridge, and attach the adapter and column reservoir.

- 11.10.5 Turn on the vacuum and begin loading the samples to the reservoir. The vacuum must be adjusted so that the flow rate is approximately 10-15 mL/min. DO NOT allow sample to go dry before the completion of the sample loading and rinsing steps.

11.10.5.1 Be certain to rotate method blank samples through each sample port on the SPE manifold.

- 11.10.6 After the entire 250 mL sample has been loaded onto the column, rinse the sample bottle with two 7.5 mL aliquots of reagent water and pour onto the column reservoir.

- 11.10.7 After the last rinsing, allow the cartridge to dry under high vacuum (10-15 inch Hg) for at least 5 minutes (leave reservoirs on at this stage).

11.10.8 After drying, turn off vacuum pump and release vacuum.

11.10.9 Set up collection tubes to the SPE manifold using 15 mL polypropylene centrifuge tubes.

11.10.10 Rinse sample bottles with 4 mL of methanol and transfer to the column reservoir onto the cartridge. Elute the analytes from the cartridge by pulling the methanol through using low vacuum such that the solvent exits the cartridge in a drop wise fashion. Repeat sample bottle-to-column reservoir rinse and cartridge elution with a second 4 mL aliquot of methanol. A total of 8 mL methanol is collected.

11.10.11 Proceed to Section 11.11 for extract concentration to 1.0 mL.

11.11 Extract Concentration

11.11.1 Concentrate the extract volume under a gentle stream of nitrogen with water bath at 60-65°C to dryness.

11.11.2 Add 900 µL of the 96:4 Methanol:Water solution, and mix the contents well using a vortex mixer.

11.11.3 Add the appropriate amount of 537-IS to all samples.

- Add 100 µL of the 0.1 µg/mL 537-IS solution for a final concentration of 10 ng on a 1.0 mL extract.

11.11.4 Mix the contents well before transferring a portion of the extracts to polypropylene autosampler vials.

- Do not transfer the entire 1 mL aliquot to the autosampler vial because the polypropylene autosampler caps does not reseal after injection. The extracts in the autosampler vial after injection is lost due to evaporation, it is not recommended to be reused for further analysis. Extracts can be stored in the 15 mL centrifuge tubes.

11.11.5 Transfer approximately 60 µL (8 – 10 drops) of the sample to a 300 µL polypropylene autosampler vial with a plastic pipet or with an automatic pipet / plastic tip. Seal the PP autosampler vial with the polypropylene screw cap.

11.11.6 The extracts in polypropylene vials are ready for LC/MS/MS analysis.

11.11.7 Seal the rest of the extract in the 15 mL PP centrifuge tube with the PP screw cap and store the extract at room temperature.

Note: If further processing of the extracts is necessary, such as re-injection or dilution, the extract should be equilibrated to room temperature and adequately vortexed to ensure contents are mixed well before proceeding.

11.12 Extract Non-Concentration (10 mL Final Volume).

This option is employed only when requested by clients for samples not used for drinking water compliance. Generally, extracts at this volume are analyzed with calibration solutions 10x lower than those used for the 1.0 mL final volume.

11.12.1 Add 0.5 mL of the 537-IS solution to the extract. 500 µL of DI water is then added to the extract and adjusted to a final volume of 10 mL with methanol.

11.12.2 Transfer approximately 60 µL (8 – 10 drops) of the sample to a 300 µL polypropylene autosampler vial with a plastic pipet or with an automatic pipet / plastic tip. Seal the PP autosampler vial with the polypropylene screw cap.

11.12.3 The extracts in polypropylene vials are ready for LC/MS/MS analysis.

11.12.4 Seal the rest of the extract in the 15 mL PP centrifuge tube with the PP screw cap and store the extract at room temperature.

Note: If further process of the extracts is necessary, such as re-injection or dilution, the extract should be equilibrated to room temperature and adequately vortexed to ensure contents are mixed well before proceeding.

11.13 Instrument Conditions for methods 537 and 537.1

11.13.1 Recommended Shimadzu operation conditions are listed below:

Table 11.13.1 Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 35°C)	Phenomenex Gemini C18 3.0 µm, 2.0 mm x 50mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	Time	%A	%B	Curve	Flow Rate mL/min.
	0	65	35	6	0.60
	0.20	28	72	6	0.60
	1.1	28	72	6	0.60
	1.5	1	99	6	0.60
	2.6	1	99	6	0.60
	2.65	65	35	6	0.60
Maximum Pressure limit = 5,000 psi					
Injection Size	2 µL (fixed amount throughout the sequence)				
Run Time	8.0 minutes				

11.13.2 SCIEX Tandem MS conditions are listed below:

Table 11.13.2 Mass Spectrometer Interface Settings (SCIEX 5500)	
MS Interface Mode	ESI Negative Ion
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	6
Declustering Potential (V)	55
Desolvation Temp	550°C
Curtain Gas (nitrogen) Flow	35 psi
Collision Gas (nitrogen) Flow	58 psi

11.13.3 SCIEX Scanning conditions are listed below:

Table 11.13.3A Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent Pot (V)	Col. Energy (V)	Declu. Pot. (V)	Cel Exit Pot (V)	Typ RT (Min)
PFBS	Native analyte	298.9 > 80	0.011	-6	-58	-55	-37	1.76
PFBS_2	Native analyte	298.9 > 99	0.011	-5	-40	-55	-12	1.76
PFHxA	Native analyte	313 > 269	0.011	-5	-12	-25	-37	2.25
PFHxA_2	Native analyte	313 > 119	0.011	-5	-12	-25	-37	2.25
13C2-PFHxA	Surrogate	315 > 270	0.011	-5	-12	-25	-38	2.25
PFHpA	Native analyte	363 > 319	0.011	-6	-12	-25	-41	2.57
PFHpA_2	Native analyte	363 > 169	0.011	-6	-12	-25	-41	2.57
PFHxS	Native analyte	399 > 80	0.011	-12	-74	-60	-43	2.59
PFHxS_2	Native analyte	399 > 99	0.011	-12	-74	-60	-43	2.59
PFOA	Native analyte	413 > 369	0.011	-6	-14	-25	-44	2.93
PFOA_2	Native analyte	413 > 169	0.011	-5	-22	-25	-12	2.93
13C2-PFOA	Internal standard (IS)	415 > 370	0.011	-6	-14	-25	-44	2.93
PFNA	Native analyte	463 > 419	0.011	-6	-14	-25	-47	3.29
PFNA_2	Native analyte	463 > 169	0.011	-6	-14	-25	-47	3.29
PFOS	Native analyte	499 > 80	0.011	-9	-108	-65	-50	3.29
PFOS_2	Native analyte	499 > 99	0.011	-5	-58	-65	-12	3.29
13C4-PFOS	Internal standard (IS)	503 > 80	0.011	-9	-108	-65	-50	3.29
PFDA	Native analyte	513 > 469	0.011	-6	-16	-25	-51	3.65
PFDA_2	Native analyte	513 > 169	0.011	-6	-16	-25	-51	3.65
13C2-PFDA	Surrogate	515 > 470	0.011	-6	-16	-25	-51	3.65
N-MeFOSAA	Native analyte	570 > 419	0.011	-7	-36	-40	-15	3.82
d3-NMeFOSAA	Internal standard (IS)	573 > 419	0.011	-7	-36	-40	-15	3.82

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent Pot (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot (V)	Typ RT (Min)
PFUnA	Native analyte	563 > 519	0.011	-7	-18	-25	-54	3.97
PFUnA_2	Native analyte	563 > 169	0.011	-7	-18	-25	-54	3.97
N-EtFOSAA	Native analyte	584 > 419	0.011	-7	-36	-50	-15	3.99
d5-NEtFOSAA	Surrogate	589 > 419	0.011	-7	-36	-50	-15	3.99
PFDoA	Native analyte	613 > 569	0.011	-5	-18	-25	-54	4.3
PFDoA_2	Native analyte	613 > 169	0.011	-5	-18	-25	-54	4.3
PFTriA	Native analyte	663 > 619	0.011	-7	-20	-25	-54	4.56
PFTriA_2	Native analyte	663 > 169	0.011	-7	-20	-25	-54	4.56
PFTeA	Native analyte	713 > 169	0.011	-2	-22	-25	-10	4.79
PFTeA_2	Native analyte	713 > 219	0.011	-7	-36	-25	-30	4.79
HFPO-DA	Native Analyte	285 > 169	0.011	-10	-6	-48	-17	2.06
13C-HFPO-DA	Surrogate	287 > 169	0.011	-10	-10	-40	-17	2.06
F53B Minor	Native Analyte	631 > 451	0.011	-10	-40	-160	-17	3.84
F53B Major	Native Analyte	531 > 351	0.011	-10	-30	-120	-17	3.23
DONA	Native Analyte	377 > 251	0.011	-10	-16	-55	-17	2.33
DONA_2	Native Analyte	377 > 85	0.011	-10	-35	-55	-17	2.33

Native Compounds	Typical Native RT (minutes)	IS analog	Typical IS RT (minutes)	Quantitation Method
PFBS	1.46	13C4-PFOS	2.91	Internal Standard
PFHxA	1.73	13C2-PFOA	2.52	Internal Standard
PFHpA	2.14	13C2-PFOA	2.52	Internal Standard
PFHxS	2.16	13C4-PFOS	2.91	Internal Standard
PFOA	2.52	13C2-PFOA	2.52	Internal Standard
PFNA	3.09	13C2-PFOA	2.52	Internal Standard
PFOS	2.91	13C4-PFOS	2.91	Internal Standard
PFDA	3.45	13C2-PFOA	2.52	Internal Standard
PFUnA	3.60	13C2-PFOA	2.52	Internal Standard
PFDoA	3.89	13C2-PFOA	2.52	Internal Standard
PFTriA	4.16	13C2-PFOA	2.52	Internal Standard
PFTeA	4.37	13C2-PFOA	2.52	Internal Standard
NEtFOSAA	3.60	d3-NMeFOSAA	3.43	Internal Standard
NMeFOSAA	3.43	d3-NMeFOSAA	3.43	Internal Standard
HFPO-DA	2.48	13C2-PFOA	2.52	Internal Standard

F53B Minor	4.37	13C4-PFOS	2.91	Internal Standard
F53B Major	3.74	13C4-PFOS	2.91	Internal Standard
DONA	2.81	13C2-PFOA	2.52	Internal Standard
HFPO-DA	2.48	13C2-PFOA	2.52	Internal Standard
F53B Minor	4.37	13C4-PFOS	2.91	Internal Standard
F53B Major	3.74	13C4-PFOS	2.91	Internal Standard
DONA	2.81	13C2-PFOA	2.52	Internal Standard

Note 1: Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

Note 2: Mobile phase modifiers other than 20 mM ammonium acetate may be used at the discretion of the analyst, provided that the retention time stability criteria is met over a period of two weeks.

Note 3: LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFASs will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAS peaks and to keep background levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times). In addition, prior to daily use, flush the column with 100% methanol for at least 20 min before initiating a sequence. It may be necessary on some systems to flush other LC components such as wash syringes, sample needles or any other system components before daily use.

12 DATA ANALYSIS AND CALCULATION

- 12.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentration using MS/MS. Concentrations were calculated by measuring the product ions listed. Other ions may be selected at the discretion of the analyst.
- 12.2 The LC/MS/MS system is calibrated using the IS technique. Calculate analyte and Surrogate concentrations using the multiple point calibration established in the initial calibration. Do not use daily calibration verification (CCV) standard to quantitate analyte in samples (unless it is a qualitative determination and is not for reporting the data such as screening).
- 12.3 Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification and poor integration.
- 12.4 PFBS, PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions specified in the method due to the linear and branch isomers of these compounds. Most PFAS compounds are produced by one of two processes. One gives rise to linear PFAS only while the other process produces both linear and branched isomers. Both branched and linear PFAS compounds can potentially be found in the environment. For the

aforementioned compounds that give rise to more than one peak, all chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in the sample must be integrated in the same way as the calibration standard and concentrations reported as a total for each of these analytes.

12.4.1 The expected retention times (RT) are established in the Chrom data processing module during the processing of the ICAL by selecting Edit>Method>Update RT. Once the retention times are established Chrom will look for a peak within +/- 0.25 min of the RT. If a peak is detected within this window of +/- 0.25 min., Chrom will assign the absolute retention time at the apex of the peak. Chrom assigns the RT to the most predominant peak within this window. As the linear peak is the predominant peak in calibration solutions for those PFAS that are calibrated with the combination of both branched and linear isomers, those PFAS require additional evaluation in the event that the branched isomer is the predominant peak in a field sample and Chrom has not positively identified the peak due to the RT shift, as the apex may now be the branched isomer

12.4.1.1 RT are updated as needed based upon evaluation of the daily CCV.

12.4.2 A technical (qualitative) grade PFOA standard is analyzed initially, after every initial calibration and when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

- 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: “_TFOA_Instrument_Date”. Example: _TFOA_A8_15Mar2019.

12.5 If the concentration of the analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.5.1 Dilute the extract with 96%:4% (vol/vol) methanol: water solution and the appropriate amount of IS added to match the original concentration. Acceptable surrogate performance should be determined from the undiluted sample extract. The resulting data should be documented as a dilution, with an increased MRL.

12.5.2 Enter the amount of IS added to the dilution into the run reagent section of the Chrom work list so the data is calculated correctly.

- 12.5.3 Document the dilution and refortification with IS on the Dilution Report Form for Method 537, LCMS-001 537. Include this form with the TALS bench sheets.
- 12.5.4 The dilution may also be performed by using a Reagent Blank (RB) with 96%:4% (vol/vol) methanol: water solution with the appropriate concentration of IS. Document the RB used on the Dilution Report Form for Method 537, LCMS-001 537.

12.6 Calculations

- 12.6.1 Peak areas are used as a measure of response.
- 12.6.2 Average Response Factor, Linear fit, or quadratic fit is used for the calibration curve. Quadratic fit is used if the response is non-linear.
- 12.6.3 The average response of the internal standard in the calibration curve is used to correct for the response of the reported analytes.
- 12.6.4 The calibration is automatically performed by the Chrom software, based on the equations:

Equation 1 Average Response Factor:

$$x = \frac{y}{RF_a}$$

Where

x = ratio of the sample concentration to the IS concentration

y = ratio of the sample response to the IS response

RF_a = average response factor

Equation 2 Linear Calibration:

$$x = \frac{(y - b)}{m}$$

Where

x = ratio of the sample concentration to the IS concentration

y = ratio of the sample response to the IS response

b = y intercept of the curve

m = slop of the curve

Equation 3 Quadratic calibration:

$$x = \frac{-b \pm \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where

a = second order coefficient (curvature)

b = first order coefficient (slope)
 c = zero order coefficient (intercept)
 x = ratio of the sample concentration to the IS concentration
 y = ratio of the sample response to the IS response

12.7 Reporting Requirements

- 12.7.1 Reporting limits and units are described in Section 1.5.
- 12.7.2 Sample results are entered into a LIMS system in accordance with current QA policies.
- 12.7.3 Footnotes and anomalies when applicable must be included in the data package and data reduction process. Exceeded holding times must be immediately communicated to the project managers and followed by an electronically filed non-conformance memo

13 METHOD PERFORMANCE

- 13.1 The laboratory must make a one time initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests is may be necessary to use more than one QC check mix to cover all analytes of interest.
- 13.2 Initial Demonstration of Low System Background - Prepare and analyze a method blank containing preservatives using the same procedure to extract and analyze samples. Confirm that the method blank is reasonably free of contamination and that the criteria in Section 9.3 are met.
 - 13.2.1 Anytime a new lot of SPE cartridge, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that the background is reasonably free of contamination.
- 13.3 Prepare and analyze four to seven replicates of laboratory control samples (LCS) fortified near the mid range of the initial calibration curve. The reagent water must contain the same preservatives (Trizma™ at 1.25g per 250 mL) used in the method.
 - 13.3.1 Initial Demonstration of Precision (IDP) – the result of the replicate analysis must be less than 20% relative standard deviation (RSD) to be valid.
 - 13.3.2 Initial Demonstration of Accuracy (IDA) – the average recovery of the replicate values must be within ± 30% of the true value to be valid.

Demonstration of Capability Criteria		
Matrix	IDP	IDA

Reagent Water containing preservatives and fortified at mid level.	20% RSD	70 to 130% Recovery
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- 13.3.3 If any analyte does not meet the acceptance criteria, then the test must be repeated. Only those analytes that did not meet criteria in the first test need to be re-evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 13.4 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.
- 13.5 The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be at or 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 policy CA-Q-S-006. MDL studies are reviewed and maintained in the Quality Assurance Department.
- 13.6 The reporting limit is validated using the procedure outlined below. The lowest calibration standard in the initial calibration and the low-level CCV must be less than or equal to the reporting limit.
- 13.7 Spike and analyze seven replicates at the target reporting limit concentration. Process all samples adding the preservative as well. Calculate the mean and standard deviation for these replicates. Calculate the Half-Range for the prediction of interval of results (HRPIR) as follows:

Equation 5 $HR_{PIR} = 3.963S$

Where S is standard deviation, and 3.963 is a constant value for 7 replicates.

- 13.8 Calculate the upper and lower limits for the Prediction of Interval Result.

Equation 6 $PIR = \text{Mean Recovery} \pm HR_{PIR}$

Equation 7
$$\text{Upper PIR} = \frac{\text{Mean Recovery} + HR_{PIR}}{\text{Fortified Concentration}} \leq 150\%$$

Equation 8
$$\text{Lower PIR} = \frac{\text{Mean Recovery} - HR_{PIR}}{\text{Fortified Concentration}} \geq 50\%$$

- 13.9 The MRL is validated if both the Upper and Lower PIR limits meet the criteria described above. If these criteria are not met, the MRL has either been set too low

and must be determined again at a higher concentration or the preparation must be carefully repeated.

14 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g. examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15 WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1 Assorted test tubes, autovials, disposable gloves. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or at the end of the day, whichever comes first, tie the plastic bag liner shut and put the lab trash into the landfill lab trash 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 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 (which ever comes first), empty the carboy into the steel flammable solvent drum in the H3 closet. When full to between four and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.3 Aqueous 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 four and six inches of the top or after no more than 75 days, move the drum to the waste collection area for shipment.

16 REFERENCES/CROSS REFERENCES

- 16.1 EPA Method 537, EPA Document # EPA/600/R-08/092 537 by J.A. Shoemaker, P.E. Grimmett, and B.K. Boutin, Version 1.1, September 2009.
- 16.2 UCMR3 Laboratory Approval Requirements and Information Document, Technical Support Center, U.S. Environmental Protection Agency, Version 2.0, January 2012.
- 16.3 EPA Method 537.1, EPA Document # EPA/600/R-18/352 by J.A. Shoemaker and D.R. Tettenhorst, Version 1.0, November 2018.

- 16.4 The HPLC Troubleshooting Wizard, LC Resource, Inc © 2006.
- 16.5 List of other SOPs cross-referenced in SOP.
 - 16.5.1 WS-QA-0041, Calibration and Calibration Check of Balances.
 - 16.5.2 WS-PQA-008, Data Recording Requirements.
 - 16.5.3 WS-PQA-003, Quality Control Program.
 - 16.5.4 WS-QA-0023, Nonconformance and Corrective Action System
 - 16.5.5 WS-QA-0006, Method Detection Limits and Instrument Detection
 - 16.5.6 WS-WI-0053, Wisconsin DNR Requirements
 - 16.5.7 CA-Q-P-003, Calibration Curves and Selection of Calibration Points

17 METHOD MODIFICATIONS

- 17.1 The concentrations of the internal standard, surrogate, and LCS spikes have been modified to allow a higher amount to be spiked during the sample preparation process. This change was made to help reduce error.
- 17.2 An optional dilution method was added (Section 12.5.4). This change was made to help reduce error.
- 17.3 An optional concentration (non-concentration) after elution option was added for non-compliance samples (Section 11.12). This change was made to help reduce error and use new instrument technology.

18 ATTACHMENTS

- 18.1 Table 1 – Method 537 Analytes, Surrogates, and Internal Standards

19 REVISION HISTORY

Revisions prior to 1/1/2019 have been removed and are available in previous versions of this SOP.

- 19.1 WS-DW-0004, Revision 2.7, Effective 05/05/2020
 - 19.1.1 Section 1.2 & Table, removed MDL and MDL values. These are available in TALS if needed.
 - 19.1.2 Added Section 1.4, referring to the Wisconsin DNR Work Instruction.
 - 19.1.3 Section 2.1, inserted “for non-compliance samples” in the last sentence.

- 19.1.4 Table 5.2, Updated Methanol NFPA rating to (2-3-0).
- 19.1.5 Note following Section 6.1, removed: “If Agilent PTFE lined screw caps are determined to be free from contamination they may be used.”
- 19.1.6 Section 6.4, changed temperature from 55 to 65 Centigrade.
- 19.1.7 Section 6.7, changed to read:” Narrow range pH test paper encompassing pH 6 to 8, Whatman part number 2629 990 or equivalent.”
- 19.1.8 Inserted Section 6.12, Vortex Mixer.
- 19.1.9 Section 7.5, removed “Methanol-Water 95.55:4.45” and renumbered the section. This reagent is not used.
- 19.1.10 Section 8.1, first sentence – removed “or larger”.
- 19.1.11 Section 8.1, third sentence, added “(pH7)” following Trizma to clarify the formulation used.
- 19.1.12 Section 8.1, Removed the last sentence.
- 19.1.13 Section 8.8, added the sentence: “File an NCM for inclusion in the final report in TALS, and ensure that it is emailed to the project manager.”
- 19.1.14 Following Section 8.11, appended the note “Note: For drinking water samples, nonconformances for items 8.9 through 8.11 will result in non-compliance results that will be rejected by the regulator.
- 19.1.15 Section 8.11.1, removed the entire item regarding possible storage as 0-6C for extracts.
- 19.1.16 Inserted Section 9.4.2, “The low concentration LCS must be no greater than 2x MRL”, and renumbered the following items.
- 19.1.17 Section 9.5.4, changed to read, “If the CCV standard fails for the surrogate compounds then recalibration and reinjection is in order.”
- 19.1.18 Section 10.5, second bullet, removed: “If a curve is used, the intercept of the curve must be less than $\pm \frac{1}{2}$ the reporting limit for the analyte.”
- 19.1.19 Added Section 10.5.1, further describing the evaluation of calibration curves.
- 19.1.20 Table 10.9, right most columns, replaced “of true value” with “from average ICAL response”.
- 19.1.21 Section 11.3.5, replaced “1” with “0.5”.

- 19.1.22 Section 11.3.10, appended to the first sentence: "...and the sample is not acceptable for this method."
- 19.1.23 Section 11.4, removed as it was redundant. Replaced with direction on creating MB and LCS aliquots.
- 19.1.24 Section 11.5 reworded and reconfigured to describe how to determine volume, and includes information formerly in the note following Section 11.10.
- 19.1.25 Removed Section 11.10.7, "After the entire sample has passed, rinse the reservoir with an additional 5.0 mL of reagent water".
- 19.1.26 Section 11.11.1, updated the temperature range from 55C to 60-65C.
- 19.1.27 Section 11.11.2, changed the reagent proportion to 96:4.
- 19.1.28 Section 11.11.7, changed the storage conditions to room temperature.
- 19.1.29 Section 11.12.4, changed the storage conditions to room temperature.
- 19.1.30 Table 11.13.3A, changed the masses monitored for HFPO-DA and 13C-HFPO-DA to 285 > 169 and 287 > 169 respectively.
- 19.1.31 Inserted Section 12.4.1, describing how retention times are assigned for components with branched isomers present.
- 19.1.32 Editorial Changes
- 19.2 WS-DW-0004, Revision 2.6, Effective 01/20/2020
 - 19.2.1 Section 7.7, appended, "Bottleware with the appropriate amount Trizma® already present may be purchased and used."
 - 19.2.2 Section 7.9, appended "Prior to use, allow all solutions to warm to room temperature. Mix using a vortex mixer prior to taking aliquots for use."
 - 19.2.3 Section 7.9.4, first sentence, inserted "Absolute Standards or".
 - 19.2.4 Table 7.9.12A: Updated final volume to 20 mL, corrected concentrations of the source reagents.
 - 19.2.5 Table 7.9.12B: Updated all target analyte concentrations to be 10x the former value.
 - 19.2.6 Section 7.9.15, appended, "Alternatively, a mixed stock solution may be used, for example part number 99896 from Absolute Standards)."

- 19.2.7 Table 7.9.15, updated volumes added.
- 19.2.8 Table 7.9.16, updated concentrations.
- 19.2.9 Section 11.9.13, changed to read, “Proceed to Section 11.11 for extract concentration to 1.0 mL.”
- 19.2.10 Section 11.9.14 removed.
- 19.2.11 Inserted Section 11.11 and following, “Extract Non-Concentration (10 mL Final Volume).”
- 19.2.12 Editorial Changes
- 19.3 WS-DW-0004, Revision 2.5, Effective 10/29/2019
 - 19.3.1 Inserted Section 3.1, cross-reference to some laboratory abbreviations.
 - 19.3.2 All references to Waters instrumentation and instrument conditions have been removed, including from Sections 6 and 11.
 - 19.3.3 Section 6.9.2, removed references to automated and robotic SPE systems.
 - 19.3.4 Inserted Section 7.6, preparation of 96:4 methanol:water.
 - 19.3.5 Section 7.8, Reagent Water – appended “i.e. < 1/3 RL”.
 - 19.3.6 Removed Section 7.9.2, reference to making stock solutions from neat materials. The laboratory purchases stock solutions.
 - 19.3.7 Sections 7.9.6, 7.9.7, 7.9.8, 7.9.9, 7.9.10, 7.9.11, 7.9.12, updated the solvent to incorporate 4% water with the methanol
 - 19.3.8 Section 7.3, Ammonium acetate holding time is shortened to 48 hours in accordance with Method 537.
 - 19.3.9 Section 11.4.1, removed “If the samples were collected in larger bottles, pour 250 mL of each sample into 250 mL propylene bottles.”
 - 19.3.10 Section 9.2, changed to read, “Batches are defined at the sample preparation step. Batches should be kept together through the analytical process, and it is mandatory to analyze prepared extracts on the same instrument.”
 - 19.3.11 Section 9.3, clarified MB acceptance criteria.
 - 19.3.12 Section 9.5.5 and 9.6.6, inserted direction to immediately notify the client in the event of surrogate failure, as that can impact data usage.

- 19.3.13 Section 9.6.7, inserted direction to notify the client in the event of internal standard failures.
- 19.3.14 Section 9.8.2, changed to read, “Analyte recoveries may exhibit matrix bias. Spiked analytes with recoveries or precision outside of the control limits may be reported if result for the LCS is in control, however, the results are considered suspect due to matrix effects and must be narrated as such.”
- 19.3.15 Inserted Section 9.8.3, “For samples fortified at mid or high levels, the analyte recoveries should range between 70-130%. For low level at or near the RL (within 2x RL) the analyte recoveries should range between 50 to 150%. Corrective actions must be documented on a nonconformance memo and implemented when recoveries of any spiked analyte are outside of the control limits.”
- 19.3.16 Section 9.9, clarified acceptance criteria.
- 19.3.17 Section 9.10.2, clarified FRB acceptance criteria.
- 19.3.18 Editorial changes.
- 19.4 WS-DW-0004, Revision 2.4, Effective 10/02/2019
 - 19.4.1 Section 1.2 revised last sentence to, “The specific compounds and their minimum detection limits (MDL) and reporting limits (RL) are indicated below.”
 - 19.4.2 Section 1.2 updated table to reflect current laboratory detection limits.
 - 19.4.3 Removed Section 1.3, “The Minimum Reporting Limit (MRL) in aqueous sample is 2 - 6 ng/L for the PFAS compounds.”
 - 19.4.4 Section 7.3 revised, “1.509 g” to “1.54 g”.
 - 19.4.5 Section 9.9 added, “The ICV must meet mid-level CCV acceptance criteria, see Section 10.9.3.”
 - 19.4.6 Section 11.4.1 revised, “25 to 30 mg” to “1.25 g”.
 - 19.4.7 Section 11.9.13 revised to, “If the extract is not concentrated first add 0.5 mL of the 537-IS solution to the extract. 500 µL of DI water is then added to the extract and adjusted to a final volume of 10 mL with methanol. Then proceed to Section 11.10.5.”
 - 19.4.8 Throughout the SOP, revised storage conditions from “2 – 6°C” to “0 – 6°C”.

- 19.4.9 Section 15.2 revised "...between two and six inches of the top..." to "...between four and six inches of the top...".
- 19.4.10 Section 15.3 revised last sentence to, "When the drum is full to between four and six inches of the top or after no more than 75 days, move the drum to the waste collection area for shipment."
- 19.4.11 Editorial changes.
- 19.5 WS-DW-0004, Revision 2.3, Effective 07/16/2019
 - 19.5.1 Added Section 9.10, "Field reagent blank (FRB) – A FRB should be submitted per sample set and extracted with the associated samples" and its associated subsections.
 - 19.5.2 Editorial changes.
- 19.6 WS-DW-0004, Revision 2.2, Effective 05/14/2019
 - 19.6.1 Section 1.2 updated table with correct CAS numbers, MRLs, and acronyms.
 - 19.6.2 Section 1.2 removed, "**Note:** Bold indicates the analytes are under the UCMR3 program."
 - 19.6.3 Section 1.2 added note, "(1) In some literature, the acronym ADONA refers to the ammonium salt, CAS 958445-44-8, and DONA refers to the parent acid. In Method 537.1, ADONA refers to the parent acid. DONA is the acronym present on the laboratory raw data."
 - 19.6.4 Section 7.8.6.1 table updated notations and stock concentrations.
 - 19.6.5 Section 7.8.6.1 revised note (2), "DONA is the laboratory abbreviation for 4,8-dioxa-3H-perfluorononanoic acid, CAS 958445-44-8. "ADONA" is used in the laboratory to refer to the ammonium salt, which is calculated by multiplying the DONA concentration by 1.048. This analyte is added as ADONA is calculated from this analyte."
 - 19.6.6 Section 12.4.1.1 revised last sentence to, "Use the following naming convention: "_TFOA_Instrument_Date". Example: "_TFOA_A8_15Mar2019."
 - 19.6.7 Section 13.5 removed reference to WS-QA-0006.
 - 19.6.8 Removed Section 13.10, "MDL fortification performed at 8.0 to 72 ng/L for instrument A6 has met criteria for Method 537A."

- 19.6.9 Throughout SOP added references to method 537.1 where applicable.
- 19.6.10 Editorial changes.
- 19.7 WS-DW-0004, Revision 2.1, Effective 01/31/2019
 - 19.7.1 Section 1.1 revised to, “This procedure is based upon EPA Methods 537 Version 1.1 (September 2009) and 537.1 Version 1.0 (November 2018).”
 - 19.7.2 Section 1.2 revised to, “This method covers the determination of selected per-fluorinated and poly-alkyl substances (PFAS) in drinking water using Liquid Chromatography with tandem Mass Spectrometry (LC/MS/MS). The specific compounds and minimum reporting limits (MRL) are indicated below.”
 - 19.7.3 Section 1.3 revised to, “The Minimum Reporting Limit (MRL) in aqueous sample is 2 - 6 ng/L for the PFAS compounds”
 - 19.7.4 Section 1.2 added HFPO-DA, F35B Minor, F35B Major, and ADONA entries to table.
 - 19.7.5 Section 7.8.6.1 added HFPO-DA, F35B Minor, F35B Major, ADONA, and DONA entries to table.
 - 19.7.6 Section 7.8.9 added 13C3-HFPO-DA entry to table.
 - 19.7.7 Section 7.8.11 added HFPO-DA, F35B Minor, F35B Major, ADONA, DONA, and DONA_2 entries to table.
 - 19.7.8 Section 7.8.11 removed note from table, “* L1 – L6 is used for UCMR3 program”.
 - 19.7.9 Section 7.8.15 added HFPO-DA, F35B Minor, F35B Major, ADONA, DONA, and DONA_2 entries to table.
 - 19.7.10 Section 9.10 removed “as needed” from item 7.
 - 19.7.11 Section 11.11.3 added HFPO-DA, F35B Minor, F35B Major, ADONA, DONA, and DONA_2 entries to first table.
 - 19.7.12 Section 11.11.3 added HFPO-DA, F35B Minor, F35B Major, and DONA entries to second table.
 - 19.7.13 Section 12.4.1 revised “an” to “every”, and removed “when a new column is installed”.
 - 19.7.14 Added Section 12.4.1.1, “Attach this document to the ICV from the associated ICAL by scanning the document and associating it to the file as
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a document type of High Res MS Tune in TALS. Use the following



TABLE 1 - Method 537/537.1 Analytes, Surrogates, and Internal Standards				
Analyte		CAS No.	Acronym	Molecular Formula (as anions)
Perfluorobutanesulfonate, potassium salt		29420-49-3 375-73-5	PFBS	C ₄ HF ₉ SO ₃
Perfluorohexanesulfonate, sodium salt		82382-12-5 355-46-4	PFHxS	C ₆ HF ₁₃ SO ₃
Perfluorooctanesulfonate, sodium salt		4021-47-0 1763-23-1	PFOS	C ₈ HF ₁₇ SO ₃
Perfluorooctanoic acid		335-67-1	PFOA	C ₈ HF ₁₅ O ₂
Perfluoroheptanoic acid		375-85-9	PFHpA	C ₇ HF ₁₃ O ₂
Perfluoronanoic acid		375-95-1	PFNA	C ₉ HF ₁₇ O ₂
Perfluorohexanoic acid		307-24-4	PFHxA	C ₆ HF ₁₁ O ₂
Perfluorodecanoic acid		335-76-2	PFDA	C ₁₀ HF ₁₈ O ₂
Perfluoroundecanoic acid		2058-94-8	PFUdA	C ₁₁ HF ₂₁ O ₂
Perfluorododecanoic acid		307-55-1	PFDoA	C ₁₂ HF ₂₃ O ₂
Perfluorotridecanoic acid		72629-94-8	PFTTrDA	C ₁₃ HF ₂₆ O ₂
Perfluorotetradecanoic acid		376-06-7	PFTeDA	C ₁₄ HF ₂₇ O ₂
N-Methylperfluoro-1-octanesulfonamidoacetic acid		NA	N-MeFOSAA	C ₁₁ H ₆ F ₁₇ NO ₄ S
N-Ethylperfluoro-1-octanesulfonamidoacetic acid		NA	N-EtFOSAA	C ₁₂ H ₈ F ₁₇ NO ₄ S
SU1	Perfluoro-n-[1,2- ¹³ C ₂]hexanoic acid	NA	¹³ C ₂ -PFHxA	¹³ C ₂ ¹² C ₄ HF ₁₁ O ₂
SU2	Perfluoro-n-[1,2- ¹³ C ₂]decanoic acid	NA	¹³ C ₂ -PFDA	¹³ C ₂ ¹² C ₈ HF ₁₉ O ₂
SU3	N-deuterioethylperfluoro-1-octanesulfonamidoacetic acid	NA	d ₅ -N-EtFOSAA	C ₁₂ D ₅ H ₃ F ₁₇ NO ₄ S
IS1	Perfluoro-[1,2- ¹³ C ₂]octanoic acid	NA	¹³ C ₂ -PFOA	¹³ C ₂ ¹² C ₆ HF ₁₅ O ₂
IS2	Perfluoro-[1,2,3,4- ¹³ C ₄]octanesulfonate, sodium salt	NA	¹³ C ₄ -PFOS	¹³ C ₄ ¹² C ₄ HF ₁₇ SO ₃
IS3	N-deuteriomethylperfluoro-1-octanesulfonamidoacetic acid	NA	d ₃ -N-MeFOSAA	C ₁₁ D ₃ H ₃ F ₁₇ NO ₄ S

naming convention: “_ZbatchnumberTPFOA”.”

19.7.15 Added Section 16.3, “EPA Method 537.1, EPA Document #: EPA/600/R-18/352 by J.A. Shoemaker and D.R. Tettenhorst, Version 1.0, November 2018.”

19.7.16 Throughout the SOP revised “PFAA” to “PFAS”.

19.7.17 Editorial changes.

	<p>Always check on-line for validity.</p> <p style="text-align: center;">Perfluorinated Alkyl Substances (PFASs) in Drinking Water by Method 537.1 Version 1.0</p>	<p>Level:</p> <p>Work Instruction</p> 
<p>Document number: T-PFAS-WI25232</p>		<p>Organisation level: 5-Sub-BU</p>
<p>Old Reference:</p>		<p>Responsible: 5_EUUSLA_PFAS_Manager</p>
<p>Version: 3</p>		<p>Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep</p>
<p>Approved by: UKL3 Effective Date 05-NOV-2019</p>		

LIMS ID

14979, 14980

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Revision Log

Revision 3	Effective Date: This version	
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reagents and Standards	Enhancement	added attachment 8 and verbiage about salt/anion concentration on CoA

Revision 2	Effective Date: 21-JUN-2019	
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Sample Collection, Preservation, and Handling	Reflect current practice	Added information about rejection of samples for out of range temperature and pH.
Apparatus and Equipment	Enhancement	Updated Glass volumetric flasks to PP volumetric flasks
Reagents and Standards	Enhancement	Changed to use of PP for all standard prep. No glass containers to be used.
Statistical Information /method performance	Enhancement	Added details to SOP from Method section 9.
Attachments 5-7	Enhancement	Changed Glass to PP. clarified volumes

Reference

1. Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), USEPA Method 537.1 Version 1, November 2018.
2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

The method is applicable for the determination of PFAS compounds in drinking water samples. The compounds analyzed in this method are listed below. The most current MDLs and LOQs are listed in the LIMS.

Analyte	Acronym	CAS#
Hexafluoropropylene oxide dimer acid	HFPODA	13252-13-6
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9 *
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1 *
4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *

*These are the CAS numbers for the free acid form of the analyte.

**DONA is the Acronym for the free acid form of this analyte.

Basic Principles

A 250-mL aqueous sample fortified with surrogates is passed through a solid phase extraction (SPE) cartridge to extract the method analytes and surrogates. The resulting solution is analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using internal standard method.

Interferences

Compounds which have similar structures to the compounds of interest, and similar molecular weights would potentially interfere. Method interferences may be caused by coeluting peaks, contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory reagent water blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to minimize method interference:

1. LC system components may contain the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK solvent frits and tubing where possible.
2. A precolumn, Phenomenex LUNA 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFASs in standards/samples from those from the LC system and mobile phases.
3. PFAS standards, extracts and samples should not come in contact with any glass containers as these analytes can potentially adsorb to glass surfaces. PFAS analytes and internal standards commercially purchased in glass ampules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be stored in polypropylene containers.
4. All equipment used for sample extraction and analysis must be meticulously cleaned. The equipment must not be covered with aluminum foil because perfluorinated carboxylic acids can be potentially transferred from the aluminum foil to the glassware.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Health advisories have been issued for both PFOA and PFOS. Each chemical must be treated as a potential health hazard, and exposure to these chemicals must be minimized. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses must be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact, and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids. Gloves and safety glasses must be worn at all times.

All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. All samples, standards, and extracts must be collected for incineration. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC is performed to meet the requirements listed in sections 9.2.3 and 9.2.4 of the method (four LFBs spiked near the midrange of the calibration, 70-130% mean recovery, and %RSD <20%). In addition, the IDOC includes the preparation (Extraction chemist) and analysis (LC/MS/MS analyst) of a 7 replicate MDL study.

The DOC consist of four laboratory control samples (or alternatively, one blind sample) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

A. Sample Collection

1. The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

2. Collect samples in 250-mL polyethylene bottles fitted with a polypropylene screw cap containing 1.25 grams of Trizma, resulting in a Trizma concentration of 5.0 g/L. Samples do not need to be collected headspace free. Keep the sample sealed from time of collection until extraction.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10°C when the samples are received at the laboratory. If samples are received with a temperature above 10°C, the samples are rejected and the client must recollect and resubmit samples to the laboratory.

2. When samples are received, a pH check is performed. The pH must be 7 ± 0.5 . This is performed by the sample storage group prior to bottles being available to the lab for analysis. If samples are received with a pH outside of the 7 ± 0.5 pH range, the samples are rejected and the client must recollect and resubmit samples to the laboratory.
3. Samples stored in the lab must be held at or below 6°C until extraction, but must not be frozen.
4. Water samples must be extracted within 14 days. Extracts must be analyzed within 28 days after extraction. Store extracts at room temperature.

Apparatus and Equipment

1. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
2. Polypropylene bottles for reagent storage: 1000ml, Fisher; Cat. No. 02896F.
3. Analytical Balance – Capable of weighing to 0.0001 g
4. Top-Loading Balance – Capable of weighing to 0.01 g
5. Solid phase extraction (SPE) cartridge, styrene divinylbenzene polymetric sorbent- Agilent Mega Bond Elut Plexa, 6 cc cartridge, 500 mg Sorbent per cartridge, Cat. No. 12259506, or equivalent.
6. SPE vacuum extraction manifold –“Resprep” 24-port manifold; Restek Corp catalogue # 26080, or equivalent.
7. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.
8. Polypropylene SPE Reservoirs, 25ml – Sigma Aldrich Cat. No. 24258-U.
9. Centrifuge – “Q-Sep 3000”; Restek Corp. Cat. No. 26230, or equivalent, capable of 3000 rpm.
10. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent
11. Auto Pipettes – Eppendorf; capable of accurately dispensing 10 μl – 1000 μl .
12. Polypropylene pipette tips: 0-200 μl . Fisher; Cat. No. 02-681-135
13. Polypropylene pipette tips: 101-1000 μl . Fisher, Cat. No. 02-707-508
14. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M
15. Vortex mixer, variable speed, Fisher Scientific or equivalent
16. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control.
17. Reagent Water Purification System: Capable of producing ultrapure “Type 1/Milli-Q”-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

18. Thermo Target PP Polyspring inserts, catalog number C4010-630P
19. Waters 9mm vial kit pack with cap and PTFE/Sil Septa, catalog number 186005660CV, or equivalent
20. Centrifuge tubes – 50 mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
21. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125
22. Syringes- Hamilton #80400 - 25 µl, #80500 - 50 µl, #80600 - 100 µl, #80700 - 250 µl, #80800 - 500 µl
23. AB Sciex Triple Quad 4500 Turbo V Ion Source, or equivalent
 - ExionLC Controller
 - ExionLC AC Pump
 - ExionLC AC Autosampler
 - Exion AC Column Oven
 - Data system –Analyst 1.6.3
24. HPLC columns
 - a. Analytical column -Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent
 - b. Pre-column- Luna 5 µm C18, 30 x 2 mm, Phenomenex Cat# 00A-4252-B0, or equivalent
25. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288, or equivalent.
26. 250ml HDPE bottle with 1.25g Trizma added, Scientific Specialties Catalog # 334008-1.25Triz.

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

- A. Reagents
 1. Methanol – Honeywell, Chromasolv LC-MS or equivalent.
 2. Milli-Q Water
 3. Ammonium acetate – Sigma Aldrich or equivalent.
 4. 20 mM ammonium acetate solution – Weigh 1.54 ± 0.01 g ammonium acetate into a 1-L bottle. Add 1 L Milli-Q water and mix well. Ammonium acetate is volatile and this solution must be replaced every 7 days or more frequently if degradation is observed. This solution may be prepared in larger or smaller volumes. Store at room temperature.
 5. 96:4 % (vol/vol) methanol: water – Add 960 mL methanol and 40 mL Milli-Q water into 1-L glass bottle, mix well. This solution may be prepared in larger or smaller volumes. Store at room temperature for 1 month.

6. Trizma Pre-set crystals - Sigma catalogue # T-7193 or equivalent, reagent grade or equivalent.

B. Standards Preparation

Standards are prepared using calibrated syringes. Polypropylene microcentrifuge tubes, polypropylene bottles, and Class A polypropylene volumetric flasks are used to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent in the volumetric flask prior to filling the volumetric to its calibration mark. Syringes should not be used to measure less than 20% of their total capacity (volume). Measurement of volumes less than 5 µl should be avoided in routine production operations. Syringes must be rinsed with clean solvent (normally methanol) prior to use and immediately after use. Calibration standards and intermediate solutions are stored at room temperature in labeled 4 ml polypropylene bottles or 15 ml polypropylene centrifuge tubes with screw caps.

Expiration dates are managed through the Standards Maintenance and Tracking (SMT). All stocks transferred from sealed glass ampoules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. All intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampoule provided by the vendor, whichever occurs sooner. Working calibration standards are given an expiration date of 2 weeks, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. Standards are prepared prior to the expiration date if degradation is observed.

Working native and labeled (surrogate and internal standard) compound spiking solutions are given an expiration date of 2 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene (PP) screw-top vials or PP centrifuge tubes at room temperature. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are stored in the SMT. Prior to use, the working spiking solution must meet recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, it should be reanalyzed in duplicate on a second LC/MS/MS system. If the reanalysis meets acceptance criteria, the solution can be used. If the reanalysis does not meet acceptance criteria, the solution must be discarded, re-prepared, and analyzed.

1. Standard Solutions and Ordering information

[Attachment 4](#) describes the required standard solutions and associated ordering information. The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable. The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See [Attachment 8](#).

If the compound purity is assayed to be 96% or greater, weight can be used without correction to calculate concentrations. Ampoules are stored in the refrigerator.

2. Standards Maintenance and Tracking (SMT) database:

In the SMT Root Name format, YY = 01-99 for year; DDD = 001 – 365 for day of the year; and ## is an incremented alpha counter.

Log purchased standards (from 1.a-g, above) into the SMT database system. Select the solution category STOCK/AMPULATED for purchased mixes and/or single-compound ampoules. The SMT system will assign formatted names to the purchased standard solutions. The automatically generated name can be overwritten with a manually created name if desired. Use labels printed through the SMT to identify and track standard solutions after transfer from original ampoule to storage vial.

3. Preparation of intermediate-concentration solution mixes is necessary to prepare the working initial calibration standards. [Attachment 5](#) describes the Intermediate solutions required for preparation of working calibration standards, ICV and linear branched standard solutions. Enter the appropriate information into the SMT database under New>>>INTERMEDIATE as the intermediate solutions are prepared.

4. All working calibration solutions are prepared in 96% methanol/water and are stable for at least 2 weeks if stored at room temperature. The working calibration standards are prepared using ampouled stocks(see [Attachment 4](#)), as well as the intermediate solutions (see [Attachment 5](#)) The preparation of the working calibration standards are described in [Attachment 6](#).

Calibration standards consist of five levels of increasing native-compound concentration and constant concentrations of mass-labeled compounds functioning as internal standards. Also included in the initial calibration are: a Method Detection Limit (MDL)-level standard, a linear and branched standard for T-PFOA, and an Initial Calibration Verification (ICV) standard. The ICV should be from an alternate vendor ("2nd source"), if possible, other than the primary source. For PFAS analysis, it is common to use mixes from the same vendor (Wellington Labs), but from a separate/different manufactured lot number.

The following represents an example of standard naming/codes generated from the SMT database for an initial 5-point (level) PFAS calibration, with MDL, linear and branched standard for T-PFOA, and ICV standards:

MDLH20X1833H (MDL)

CALH2011833H (CAL1)

CALH2021833H (CAL2)

CALH2031833H (CAL3)

CALH2041833H (CAL4)

CALH2051833H (CAL5)

ICVH20X1833H (ICV)

LBH20X1833H (L+B Standard)

5. Preparation of working native spike solutions (for spiked batch QC; LFB/LFBD; LFSM/LFSMD) are described in [Attachment 7](#).

Preparation of Glassware

Not applicable

Calibration

See Procedure section B.4 through B.5.

Procedure

A. Sample Extraction

1. Weigh full sample container on a calibrated top loading balance and record the first reading in the automated prep entry system.
2. Use a 250 ml HDPE bottle pre-preserved with 1.25 grams of Trizma for the extraction blank and the LFB. Fill each bottle with 250 grams of Milli-Q water.
3. Assemble the SPE extraction apparatus and attach the SPE cartridges. Label each cartridge with the appropriate sample number.
4. Condition each SPE cartridge with 15 mL methanol followed by 18 mL of Milli-Q water. Discard the eluent. Add 4-5 mls of reagent water to each cartridge. Do not let the cartridge go dry at any point during the conditioning process.
5. Vortex all spike solutions prior to use.
6. Spike QC samples (LFB/LFBD/LFSM/LFSMD) with 40 µl of native spike. Rotate the native spike for each batch prepped between the low level, mid level, and high level spikes. Spike QC and all samples with 10 µl of surrogate spike. Vortex to thoroughly mix.
7. Attach a 25 mL SPE adaptor to each cartridge. Load the spiked samples/QC to the respective cartridges. Allow full volume to pass the each cartridge by gravity, if possible. Apply light vacuum if necessary. The flow rate should be approximately 10-15 mls per minute.
8. After the sample has fully eluted, rinse the sample bottle with 7.5 mls of Milli-Q water and add to the cartridge. Rinse the sample bottle with a second 7.5 mls of Milli-Q water and add to the cartridge.
9. After full volume and water rinses have passed through the cartridges, discard all waste from the reservoir.
10. Wipe each SPE needle with a Kim-wipe/methanol.
11. Dry cartridges with vacuum. No more than 15" Hg for approximately five minutes.
12. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.
13. Add 4 mL of methanol to each empty sample bottles and shake well.
14. Transfer the methanol from the bottles to the SPE reservoir.
15. Repeat steps 13 and 14 a second time.
16. Elute each cartridge with the 8 mL of methanol. Collect all 8 mLs into polypropylene centrifuge tubes.
17. Concentrate on the N-Evap at no more than 40°C to dryness.

18. Add approximately 0.5ml of 96% methanol/water to the extract and then add 10 µl of internal standard mix to each extract. Reconstitute to 1.0 ml with 96% methanol/water. Extracts should be stored at room temperature in polypropylene centrifuge tubes until analysis.

19. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

B. LC/MS/MS Analysis

Tuning and calibration for the LC/MS/MS: Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference, and may not need to be changed unless loss of response is noted.

1. Chromatographic conditions

Below are the recommended chromatographic conditions for the reversed-phase separation. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

Sciex LC system Injection Volume = 1.00 µL

Stop time = 11.00 min

Flow = 0.4000 mL/min

Maximum Pressure Limit = 6500 psi

(Flow Program)

A = 20 mM ammonium acetate in water

B = 100% methanol

Time (min)	Flow (mL/min)	A. Conc (%)	B. Conc (%)
0	0.4000	95.0	5.0
1.10	0.4000	95.0	5.0
2.70	0.4000	40.0	60.0
6.30	0.4000	5.0	95.0
10.00	0.4000	5.0	95.0
10.20	0.4000	95.0	5.0
11.00	0.4000	95.0	5.0

Sampling Speed = 5.0 µL/s

Cooler Temperature = 15 degrees C

Rinsing Speed = 35 µL/S

Rinsing Volume = 500 µL

Purge Time = 25.0 min

Oven Temperature = 30 degrees C

MRM Detection Window = 60 sec

Target Scan Time = 0.6 sec

Integrated Valco Valve Method (Divert Valve Setup)

- 0 min – Waste
- 3.5 min – MS
- 10.2 min – Waste

2. Acquisition method: See [Attachment 1](#)

3. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Allow Methanol to flush for approximately an hour and then analyze five solvent blanks to clean the instrument prior to sample acquisition. An example sequence would be:

Initial Calibration Sequence:

- 1. Solvent
- 2. Solvent
- 3. Solvent
- 4. Solvent
- 5. MDL
- 6. CAL1
- 7. CAL2
- 8. CAL3
- 9. CAL4
- 10. CAL5
- 11. Solvent
- 12. ICV
- 13. L+B CAL3
- 14. CCC-CAL3

If the initial calibration passes, schedule a solvent blank followed by batch QC and samples.

Sample Sequence:

- 1. Solvent
- 2. Solvent
- 3. Solvent
- 4. Solvent
- 5. CCC1-CAL1
- 6. Method Blank (LRB)
- 7. LFB
- 8. LFBD
- 9. LFSM
- 10. LFSMD
- 11. Sample
- 12. Sample
- 13. Sample

14. Sample
15. Sample
16. CCC2-CAL3

CCC's are acquired after every 10 samples and vary from low (CAL1), mid (CAL3) and high (CAL5)

Solvent = 96% methanol in water

If the system is acquiring data overnight, schedule four solvent blanks at the end of the sequence prior to the system going into standby mode

4. Initial Calibration

- a. Inject a minimum of 5 calibration standards. The low concentration standard must be at or below the MRL (See [Attachment 3](#)). The curve must be forced through zero and may be concentration weighted 1/x.
- b. Back calculated concentrations for each analyte in each calibration level must be within 70% to 130% of its true value with the exception of the low calibration standard, CAL 1, where the back calculated concentration must be within 50% to 150% of its true value.
- c. The relative percent difference (RPD) between the high and low areas for each internal standard must be < 20%.
- d. Analyze a Linear and Branched-standard that contains linear and branch chained isomers of PFOA. The analysis of this standard is used to demonstrate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating these compounds in samples.
- e. Peak asymmetry factor: Must be calculated with each ICAL. The factor for the first two eluting peaks in the mid-level CAL standard must fall in the range of 0.8-1.5.
- f. See [Attachment 2](#) for relationship between injection standard, extraction standard, and native compound.

5. Calibration confirmation by second source standards

Once the calibration curve has been established, analyze second source mid-level standard as QCS to confirm the validity of the calibration curve/standard. A different lot of the standard or standard from a second vendor could be used. The calculated amount for each analyte must be $\pm 30\%$ of the true value.

6. Continuing calibration check

- a. The continuing accuracy must be verified by analysis of a continuing calibration Check (CCC) standard up to every ten samples and at the beginning and the end of each group of analyses. The opening CCC of the sequence must be at or below the MRL (See [Attachment 3](#)) in order to verify instrument sensitivity prior to sample analysis. All subsequent CCCs should alternate between the medium and high concentration CAL standards.
- b. The absolute areas of the quantitation ions for the internal standards (IS) must be within 70%-140% of the areas measured in the most recent CCC and within $\pm 50\%$ of the average areas measured during the most recent ICAL.

- c. The calculated amount for each target analyte and surrogate must be within $\pm 30\%$ of the true value for all CCCs except the low concentration CCC. For the low concentration CCC, each target compound must be within $\pm 50\%$ of the true value and each surrogate percent recovery must be within $\pm 30\%$ of the true value.
- d. Samples that are not bracketed by acceptable CCC runs must be reanalyzed. If the CCC recoveries are running high indicating increased sensitivity, and no detections of target analytes are observed, the data may be reported with a comment.

7. Sample analysis

- a. After the initial calibration, inject a solvent blank to demonstrate that there is no carryover issue. Usually the LFB and matrix spike samples are analyzed at the beginning of the analytical set, samples are analyzed next. Bracket each set of up to ten samples with a continuing calibration Check (CCC) standard.
- b. Process each sample and review the chromatogram closely. Evaluate all integrations, baseline anomalies, and retention time differences.
- c. All internal standard recoveries in QC and field samples must be within 70%-140% of the response in the most recent CCC and within $\pm 50\%$ difference of the average response from the most recent ICAL. If the internal standard areas do not meet these criteria, a second aliquot of the sample may be analyzed. If the analysis of the second aliquot is acceptable, report those results. If the analysis of the second aliquot still yields internal standard responses that do not meet criteria, the sample may need reextracted if it is still within holding time or flagged with a comment on the analysis report.
- d. All surrogate recoveries in QC and field samples must within the range of 70%-130%. If the recoveries fall outside this range the sample must be re-extracted.
- e. Evaluate laboratory reagent blank (LRB). No target analytes can be detected above the MDL, which is less than 1/3 the MRL. If there are positive detections in the LRB but no detections in the associated samples the data may be reported. If there are positive detections in the LRB above the MDL, and detections of the same target analytes in the associated samples, the samples must be re-extracted.
- f. Evaluate the laboratory fortified blank (LFB). All native recoveries should be within 70%-130% except the low fortified LFB. The acceptance criteria for the low fortified LFB is 50%-150%. If recoveries fall outside these acceptance ranges for the LFB (native recoveries), reinject all samples with the LFB. If issue persists, further evaluation of the system and possible re-extraction may be required. If re-extraction is required, all associated samples must also be re-extracted.
- g. Evaluate the laboratory fortified sample matrix and matrix duplicate(LFSM/LFSMD). All native recoveries should be within 70%-130% except the low fortified LFB. The acceptance criteria for the low fortified LFB is 50%-150%. The RPD's should be $\leq 30\%$.
- h. If any targets are detected above the reporting limit in a sample, evaluate the field reagent blank (FRB). If any targets found in the field samples are also found in the FRB at concentrations $> 1/3$ the MRL, all field samples associated with the FRB must be recollected. and reanalyzed. If a FRB is not submitted with a field sample, a comment will be added to the analysis report. The FRB must contain the same lot # of Trizma as the associated sample set.

Calculations

1. Internal standards

Calculating the %D

%D for CAL standards = $((\text{IS Area} - \text{AVG Area from the Calibration}) / \text{AVG Area from the Calibration}) * 100$

For samples:

%Recovery IS = $((\text{IS Area} - \text{IS area CCC}) / \text{IS Area CCC} + 1) * 100$

Where CCC = most recent/opening bracket CCC

2. Surrogate Standards; Target Compounds

Combo factor = Dilution factor * Prep factor * (Sample Volume/Sample Weight)

Note: Prep factor = 1

SUR Actual Concentration = Expected Concentration (for a sample with a final volume of 1 mL) * Combo factor

Calculated Concentration = (Area Ratio/Slope of the curve) * IS Conc * DF

For surrogates: Slope of the curve = Average area from the calibration standards

IS Conc varies depending on the associated IS: 13C2-PFOA = 10 ng/mL, 13C4-PFOS = 28.68 ng/mL, d3-NMeFOSAA = 40 ng/mL.

Sample Result = Calculated Concentration * Combo factor

% REC for surrogates = $[(\text{Sample Result} - \text{SUR Actual Concentration}) / \text{SUR Actual Concentration} + 1] * 100$

See [T-PEST-WI9847](#) for details on all calculations/equations used to evaluate the initial and continuing calibration and QC samples.

Statistical Information/Method Performance

The LFB should contain all compounds of interest. LFB, MS/Ds, surrogate standard recoveries and RPD are compared to the limits stored on the LIMS. These limits are defined in the method. Historical data for MS/Ds, LFD/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

The initial demonstration of capability for this method has been carried out as listed in Section 9.2 of the reference method. See below for items not addressed elsewhere in the SOP.

1. Initial Demonstration of Low System Background - Performed any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets and autosampler vials are used.

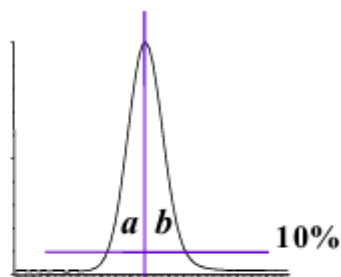
a. No peaks are present within the retention time window of any analyte that would prevent the determination of that analyte. If any peaks are present, determine the source of the contamination and eliminate the interference before sample analysis.

b. Background from method analytes must be below 1/3 of the MRL.

2. Initial Demonstration of Peak Asymmetry factor- Performed during the IDC and every time a new calibration curve is generated.

a. Calculate the peak asymmetry factors for the first two eluting peaks in a mid-level CAL standard using the following equation:

$$A_s = \frac{b}{a}$$



where:

A_s = peak asymmetry factor

B = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex

a = the width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

b. Peak asymmetry factors must fall in the range of 0.8 to 1.5

c. If the criteria are not met, corrective action must be taken prior to sample analysis.

3. Minimum Reporting Level (MRL) confirmation

a. Fortify, extract, and analyze seven replicate LFBs at the proposed MRL concentration.

b. Calculate the mean measured concentration and standard deviation of the replicates.

- c. Determine the Half Range for the prediction interval of results (HR_{PIR}) using the equation below:

$$HR_{PIR} = 3.963s$$

where

s = the standard deviation

3.963 = a constant value for seven replicates.

- d. The Upper PIR limit must be \leq 150% recovery using the equation below:

$$\frac{\text{Mean} + HR_{PIR}}{\text{Fortified Concentration}} \times 100\% \leq 150\%$$

- e. The Lower PIR Limit must be \geq 50% recovery using the equation below:

$$\frac{\text{Mean} - HR_{PIR}}{\text{Fortified Concentration}} \times 100\% \geq 50\%$$

- f. The MRL is validated if both the Upper and Lower PIR limits meet the criteria
- g. If the criteria is not met, the MRL is too low and must be determined again at a higher concentration.

Quality Assurance/Quality Control

For each batch of samples extracted, an LRB, an LFB (Milli Q water spiked with all compounds to be determined carried through the entire procedure), and an LFSM/LFSMD must be extracted. If there is limited sample that prevents the preparation of an LFSM/LFSMD then an LFBD may be prepared instead. However, the final report must then include a comment indicating the method specified LFSM/LFSMD was not analyzed due to insufficient sample submission. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. A field reagent blank (FRB) must be analyzed for each set of client samples submitted. This is to ensure no PFAS compounds are being introduced in the field. If one is not submitted, a comment will be added to the analysis report.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
 Attachment: [Attachment 1 - Acquisition Parameters \(pdf\)](#)
 Attachment: [Attachment 2 - IS-SS-Target Compound Associations \(docx\)](#)
 Attachment: [Attachment 3 - MRLs \(doc\)](#)

Attachment: Attachment 4 - Ampuolated Standards (doc)
Attachment: Attachment 5 - Intermediate Solutions (doc)
Attachment: Attachment 6 - Working Calibration Standards (doc)
Attachment: Attachment 7 - Spiking Solutions (doc)
Attachment: Attachment 8 - Example CofA (pdf)

Attachment:

Attachment 1 - Acquisition Parameters
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End of document

Version history

Version	Approval	Revision information
1	25.APR.2019	
2	21.JUN.2019	
3	28.OCT.2019	

Appendix B

PFCs Sampling Checklist

PFCs Sampling Checklist

Date: _____

Weather (*temp./precipitation*): _____ Site Name: _____

Field Clothing and PPE:

- No clothing or boots containing Gore-Tex™
- All safety boots made from polyurethane and PVC
- No materials containing Tyvek®
- Field crew has not used fabric softener on clothing
- Field crew has not used cosmetics, moisturizers, hand cream, or other related products this morning
- Field crew has not applied unauthorized sunscreen or insect repellent

Field Equipment:

- No Teflon® or LDPE containing materials on-site
- All sample materials made from stainless steel, HDPE, acetate, silicon, or polypropylene
- No waterproof field books on-site
- No plastic clipboards, binders, or spiral hard cover notebooks on-site
- No adhesives (Post-It Notes) on-site

- Coolers filled with regular ice only. No chemical (blue) ice packs in possession

Sample Containers:

- All sample containers made of HDPE or polypropylene
- Caps are unlined and made of HDPE or polypropylene

Wet Weather (as applicable):

- Wet weather gear made of polyurethane and PVC only

Equipment Decontamination:

- "PFC-free" water on-site for decontamination of sample equipment. No other water sources to be used.
- Alconox and Liquinox to 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

If any applicable boxes cannot be checked, the Field Lead shall describe the noncompliance issues below and work with field personnel to address noncompliance issues prior to commencement of that day's work. Corrective action shall include removal of noncompliance items from the site or removal of worker offsite until in compliance.

Describe the noncompliance issues (include personnel not in compliance) and action/outcome of noncompliance:

Field Lead Name: _____

Field Lead Signature: _____ Time: _____

PFC Sampling – Prohibited and Acceptable Items

Prohibited	Acceptable
Field Equipment	
Teflon® containing materials	High-density polyethylene (HDPE) materials
Low density polyethylene (LDPE) materials	Acetate Liners
	Silicon Tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
	Sharpies®, pens
Post-It Notes®	
Chemical (blue) ice packs	Regular ice
Field Clothing and PPE	
New cotton clothing or synthetic water resistant, waterproof, or stain-treated clothing, clothing containing Gore-Tex™	Well-laundered clothing made of natural fibers (preferable cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex™	Boots made with polyurethane and PVC
Tyvek®	Cotton clothing
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	<p>Sunscreens - Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are “free” or “natural”</p> <p>Insect Repellents - Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellent, Herbal Armor, California Baby Natural Bug Spray, BabyGanics</p> <p>Sunscreen and insect repellent - Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion</p>
Sample Containers	
LDPE or glass containers	HDPE or polypropylene
Teflon-lined caps	Unlined polypropylene caps
Rain Events	
Waterproof or resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
Equipment Decontamination	
Decon 90®	Alconox® and/or Liquinox®
Water from an on-site well	Potable water from municipal drinking water supply
Food Considerations	
All food and drink, with exceptions noted on right	Bottled water and hydration fluids (i.e, Gatorade® and Powerade®) to be brought and consumed only in the staging areas

Appendix C

Data Collection Sheet

Corporate Remediation Group – Residential Sampling Field Book

Site: _____ Event: _____ Date: _____ Time: _____

Personnel: _____ Project Manager: _____

Residential IDs	Well Depth	Location of Sample Port/ Amount Purged	Time sample Collected	Comments
1) _____	_____	_____	_____	_____
2) _____	_____	_____	_____	_____
3) _____	_____	_____	_____	_____
4) _____	_____	_____	_____	_____
5) _____	_____	_____	_____	_____

Location	1	2	3	4	5		
pH							
Temperature (°C)							
Specific Conductance (umho)							
Dissolved Oxygen (mg/l)							
Turbidity							
Color							
Odor							
Water Meter reading							
Time parameters collected							

Sample Date: _____ Sample Method: 1) _____ 2) _____ 3) _____ 4) _____ 5) _____

Analysis	Volume (ml)	#	Preservative	Zero HS	Additional Comments:

Analyst Name: _____ Date: _____
 Analyst Signature: _____ 04451

Appendix D

Chain-of-Custody SOP

Corporate Remediation Group Chain-of-Custody Standard Operating Procedure

Purpose

This standard operating procedure (SOP) establishes the Corporate Remediation Group (CRG) chain-of-custody (COC) standard for tracking samples from the field to the laboratory. An essential part of any sampling/analytical scheme is ensuring the integrity of the sample from collection to data reporting. The possession and handling of samples should be traceable from the time of collection through analysis and final disposition. (SW846, Chapter 9, Section 9.2.2.7).

General Information

The COC is a legal document/record that must include facility name, facility address, sample identification, dates and times of collection of samples, matrix of the sample, and details of possession (signatures of the personnel involved in the possession of the sample, including the dates of possession).

The COC also typically includes the sample analysis request, which may include laboratory name, laboratory address, contact person name/telephone number, requested analysis, number of bottles, sample preservation, reporting instructions, project or sampling event name, and field information.

Objectives of Using the COC

The objectives of using a COC are to demonstrate the chain of possession of the samples and order services from the laboratory. The following items will facilitate meeting these objectives:

- A COC must accompany every sample delivery to a laboratory, regardless of whether samples are shipped via commercial carrier, transported via laboratory courier, or hand-carried to the laboratory by the sampling team.
- Every field sample must be assigned a field sample identification number (FSID), and that FSID must be on an associated COC.
- The COC is specific to each shipping cooler. Every field sample in a cooler must have a FSID on a COC in that cooler.
- The COC must be legible and accurate.

Procedures for Completing the COC

The policy is to use either Option A or Option B as stated below.

Option A (Pre-Printed COC originated by Laboratory Personnel)

This is the preferred method for initiation of the COC, originated at the laboratory with pre-determined FSID and other requested fields. See Figure 1 for an example of COC Option A.

Laboratory personnel will do the following:

- Originate the pre-printed COC by relinquishing the bottles with a signature. The pre-printed COC contains the following information: header information (e.g., facility name, facility address, facility supervisor, project name), FSID (e.g., 2H14GWMON-MW1), sample

depths (if applicable), sample type, volume, preservative (if applicable), quantity, bottle type, method, and/or analyte.

- If the field sample IDs are known at the time of bottle preparation, pre-print FSID (e.g., 2H14GWMON-MW1) on the COC. If the FSIDs are **not** known at the time of bottle preparation, leave the FSID blank.

The project team may request that only one sample location be entered on a COC form. This has the benefit of allowing the field team to collect the samples in any order that they choose and will facilitate shipping samples from the site the day that they are collected.

Field personnel will do the following:

- If a sample is pre-printed on the COC but will not be collected:
 1. Cross out the sample on the COC.
 2. Date and initial the cross-out and identify the reason on the COC (e.g., well is dry).
- If an extra sample is collected that was not pre-printed on the laboratory relinquished COC, add this sample to a separate blank COC (not the COC that was relinquished by laboratory personnel).
- If all of the samples listed on the laboratory relinquished COC cannot be collected in one day, use Option B.

Option B (Pre-Printed/Blank COC Originated in the field)

- Laboratory personnel issue COC forms with the bottles. These forms can be pre-printed or left blank.
- Field personnel will do the following:
 1. Collect the samples and write the FSID on the COC.
 2. Write the date and time of sample collection on the COC.
 3. Enter the remaining information on the COC [i.e., sample type, volume, preservative (if applicable), quantity, bottle type, method, and/or analyte (if not already pre-printed on the COC)].
 4. Once the samples are ready to be shipped to the laboratory and all of the aforementioned information has been entered for the samples collected, relinquish the samples to the laboratory with his/her signature, date, and time (see Figure 2 for examples of Option B).

Signatures

Option A

If laboratory personnel initiate the COC:

1. Laboratory personnel relinquish the bottles with a signature.
2. Field personnel receive the cooler(s) from the courier (i.e., Laboratory/Federal Express/UPS). Field personnel will sign for the shipment if received directly from a courier.
3. Field personnel:
 - Check contents of cooler against COC.
 - Sign the COC in the "Received By" box.
 - Relinquish the samples to the laboratory once they have finished sampling.

4. Laboratory personnel:
 - Cross-out the unused “Received By/Relinquished By” boxes prior to signing.
 - Sign the COC upon receipt of the samples.
5. Field personnel file and keep the Federal Express/UPS bill of lading **to** and **from** the site (if used).

Option B

If laboratory personnel did not initiate the COC:

1. Field personnel sign the COC upon completion of sampling in the Relinquished By box.
2. Laboratory personnel sign the COC upon receipt of the samples and cross-out the unused “Received By/Relinquished By” boxes.
3. Field personnel file and keep the Federal Express/Airborne bill of lading **from** the site (if used).

“Cross Outs” on COC

- If corrections are made to the COC while in the field, field personnel must date and initial the item that was crossed out.
- If corrections are to be made to the COC **after** it has left the field, Analytical Data Quality Management (ADQM) personnel:
 1. Document the error. This can be an email between the project team and ADQM or other written communication.
 2. Mark up the COC field copy. This can be done by either ADQM or the project team. All of the corrections must be dated and initialed.
 3. Send an email with the reason for the correction and the corrected COC to the person requesting the correction (if other than ADQM personnel) for signature.
- Once the requestor has reviewed the documentation, he/she sends an email acknowledging the correction back to ADQM personnel with a signature on the corrected COC.
- ADQM keeps the original with the file and sends a copy to the laboratory and to the project manager.

Trip Blank Collection Date and Time

The trip blank for volatile organic compounds (VOCs) is originated in the laboratory and sent to the field with the sample bottles for field collected VOCs. The laboratory does not add a date and time for the collection of the VOC. However, the Locus EIM database requires both date and time for all field samples. Therefore, field personnel will use the date and time of the first collected VOC as the sample collection time for the trip blank.

Location of COC With Respect To Cooler

Laboratory personnel:

1. Print the COC on thermal paper (or duplicate copies) so that all parties handling the samples can maintain a copy in their files.

2. Place all copies of original COC or form (which will become a COC once a signature has been added) inside a zip-lock plastic bag and pack inside the top of the cooler when shipped to the field.

Field personnel:

1. Place the original COC and laboratory copy inside a sealed zip-lock plastic bag and pack in the top of the cooler containing the samples listed on that COC. The zip-lock bag may also be taped to the inside of the cooler lid.
2. Keep one copy of the COC for their files.

Bottle Labels

Field personnel must make sure that the bottle label contains the FSID, the preservative added, the number of bottles, the analyses, and whether or not the sample is filtered. The information on the bottle label must match the information on the COC.

Date/Time of Sample Collection

Field personnel must:

1. Write the date on COC as MM/DD/YY (e.g., 08/31/14).
2. Write the time on COC in 24-hour or military time (e.g., 13:30). The time of collection is recorded as the time the sample was initially taken. A separate time of collection is not required for each parameter (e.g., time for volatiles, time for semi-volatiles, etc.) The date and time of collection of field duplicate samples, and matrix spike/matrix spike duplicate samples must be the same date and time as the original sample.

Custody Seals

Laboratory personnel include custody seals with each cooler shipment.

Field personnel:

1. Pack the samples on ice in the cooler. It is recommended that a large heavy plastic bag be used to enclose all samples, ice, and packing material. The bag should be sealed prior to enclosing the zip-lock bag with the COC form.
2. Once the cooler is ready for shipment, tape the custody seals to the broad side of the cooler lid opposite the hinges in such a way that the seals will be broken if the cooler is opened.
3. Sign and date the custody seals prior to shipment to the laboratory. If field personnel break the seals of the cooler prior to shipment (e.g., to re-ice the samples), field personnel must attach another set of seals to the cooler with the field personnel's signature and the date.
4. If specified in the QAPP, attach custody seals to the bottles. Place the seal over the cap of the bottle and down both sides in such a way that, if the cap is unscrewed, the seal will be broken.

Cooler Numbers

ADQM personnel may instruct the laboratories to write cooler numbers on coolers and associated COC forms containing samples to be analyzed for volatiles (e.g., label attached with cooler number or cooler number written directly on cooler).

Special Requests/Concerns

Field personnel should use comment section of the COC for special requests/concerns such as “analyze within 7 days” and “high field PID readings.”



Lancaster
Laboratories

Analysis Request / Environmental Services Chain of Custody

For Lancaster Laboratories Use Only

Group No.: _____ Sample Nos.: _____

Acc't: 07032 SF: 69273 SCR No.: 137632 Cooler No.: _____

25201

Cooler Temperature upon receipt: _____ °C Container No.: _____

Facility Name:		Project Manager:					Analyses Required											Comments:									
Facility Contact:		Facility Contact Phone No.:					GC/MS Semivolatiles (8270)	Alkalinity (2320 B)	Br-, Cl-, F-, SO4 (300.0)	PPL Metals + Al, Ca, Fe, Mg, Mn, K, Na	NH3 (4500 NH3 B&D)	NO2 (353.2)	NO3 (353.2)	pH (4500-H+B)	S2- (4500 S2 D)	TOC (SW-846 9060)	TP as PO4 (EPA 365.1)		PPL Metals + Fe, Mn								
Facility Address:		Job No.:																									
		Release No.:																									
		PO Number: LBIO-																									
Sampler(s):																											
Project Name: GROUNDWATER SAMPLING 4/16																											
Sample Identification			Date Collected	Time Collected	Matrix	Containers												Condition upon receipt:									
			Volume (ml)	Preserv	No.																						
EA0416-J04-M02E					WW	250	HNO3	1																			
EA0416-J04-M02E					WW	1000	H2SO4	1																			
EA0416-J04-M02E					WW	250	None	2	X																		
EA0416-J04-M02E					WW	250	H2SO4	1																			
EA0416-J04-M02E					WW	250	NaOH/ZnAc	1																			
EA0416-J04-M02E					WW	40	H2SO4	1																			
EA0416-J04-M02E					WW	250	None	1			X																
EA0416-J04-M02E					WW	40	None	1																			
EA0416-J04-M02E					WW	250	None	1				X															
EA0416-J04-M02E					WW	40	H3PO4	5																			
EA0416-J04-M02E-Z					WW	250	HNO3	1																			
Turnaround Time Requested (please circle) : <u>Standard</u> RUSH Number of days: <u>8</u>									Special Instructions: 48 Hour Holding Time for nitrite (EPA 353.2)																		
Bottles Relinquished by:			Date		Time		Bottles Received by:			Date:		Time:															
Bottles Relinquished by:			Date		Time		Bottles Received by:			Date:		Time:															
Bottles Relinquished by:			Date		Time		Bottles Received by:			Date:		Time:															
Bottles Relinquished by:			Date		Time		Bottles Received by:			Date:		Time:															

Appendix E

Shipping SOP

Sample Packing and Shipping Standard Operating Procedure

Scope of Application

This Standard Operating Procedure describes the protocols for properly packaging and shipping environmental field samples to an off-site laboratory for analysis. These procedures have been developed to mitigate the risk of damage to the samples, ensure the maintenance of samples temperature within the cooler, and prevent the spillage of the sampled materials should a container be broken. The procedures described do not include the selection of sample containers and preservatives, which may vary depending on the analytical method and project requirements, and should be addressed in the project-specific Quality Assurance Project Plan or Work Plan.

Materials and Equipment

- Packaging and strapping tape
- Scissors
- Laboratory address and custody seal labels
- SDS
- Chain of Custody (COC) record
- Sample containers
- Shipping Coolers (Coleman or other sturdy, waterproof cooler)
- Ice
- Cooler liner bag
- Bubble wrap
- Ziploc bags

Related Procedures

Refer to *Standard Operating Procedure for Chain-of-Custody*.

Procedures for Environmental Samples

Environmental samples are defined as those samples collected from environmental matrices such as soil, groundwater, surface water, or sediments. Environmental samples should be packaged for shipment as follows.

Prior to Sample Collection

1. Remove all empty sample containers from the storage/shipping cooler and place on a clean surface.
2. Unroll the plastic liner bag and spread out the corners.
3. Place bubble wrap on cooler bottom.
4. Place bag in the cooler on top of bubble wrap.

5. Place the empty sample containers, temperature blank, and trip blanks (if applicable) in the bag.
6. Place one unopened bag of ice on the empty sample containers. As samples are collected, they will be returned to the iced cooler for holding prior to shipment.
7. After sample containers are filled, check the pH of aqueous samples with litmus paper (place small amount of sample into a Dixie cup, then discard cup and contents following measurement). Do not open or test the pH on sample containers with zero headspace requirements (i.e., VOA vials). If you have any questions on what the pH of the sample should be, ask the project chemist.

Following Sample Collection

1. Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).
2. Verify that all samples are present, properly labeled, and securely sealed.
3. Compare information on sample container labels with information on completed chain-of-custody record.
4. Drain any water from inside the cooler liner bag and repack sample containers inside.
5. Place the temperature blank as close to the center of the cooler as possible. This assures that a representative measurement of the sample temperatures upon laboratory receipt.
6. Place sample containers in an upright position. Do not over pack them. Allow sufficient space between them to permit continuous cooling and prevent breakage.
7. Pack the cooler liner bag with ice, making sure bag corners are full of ice.
8. Fill the cooler to just above the top of the tallest sample container.
9. If the cooler contains **only** 40 ml VOA vials, do not fill extra space in cooler with ice.
10. Use extra bubble wrap as filler and add 2 large Ziploc bags filled with ice to keep samples chilled. The sample vials should not come into direct contact with the ice to prevent freezing and possible cracking of the sample containers.
11. Large coolers (54-68 quart) require 3 to 4 bags of ice and medium coolers (48 quart) require 2 to 3 bags. When shipping for overnight delivery or when the ambient air temperature is warm, extra care must be taken to ensure that the cooler is completely packed with ice. During periods of extreme hot weather, particularly when sampling surface soils or standing water, it may be necessary to pre-chill the samples prior to packing for shipping, or to pack fewer samples per cooler to allow for sufficient cooling.
12. Remove as much air as possible and twist liner bag until the twisted section is approximately one foot long.
13. Wrap packing tape at the base of the twisted section until secure, then “goose neck” the section by twisting and folding it back on itself and securing again with the wrapping tape.
14. Place the completed chain-of-custody records and SDS forms inside a Ziploc bag on top of the cooler liner bag. The Ziploc must be sealed and facing toward the cooler lid.
15. Close and latch the cooler lid.
16. Remove all labels from the outside container.

17. Using clear strapping or packing tape, wrap the tape around several times approximately 3-4 inches from the edge of the cooler.
18. Apply signed and dated lab custody seal to the tape over the cooler lid across opening.
19. Adhere laboratory address label on top of lid and over tape.
20. Wrap tape around cooler and over lab custody seal and address label and at least two times completely around.
21. Repeat on opposite side with the remaining lab custody seal.
22. Samples that are not required to be kept chilled (i.e., grain size or other geotechnical samples) should be packed according to the procedure outlined above, omitting the ice and securing the samples with bubble wrap or other inert packaging material.
23. The person responsible for coordinating the shipment of samples to the laboratory should be aware of any weight restrictions or other delivery or policy limitations prior to transfer of the coolers to the courier.

Procedure for Non-Environmental/Hazardous Samples

Non-environmental or hazardous samples are defined as those that are typically highly contaminated, such as oils (LNAPL and DNAPL), discarded products, wastes, and other materials. The procedures for packaging, labeling, and shipping hazardous or non-environmental samples vary depending on the material involved and method of shipment.

Department of Transportation (DOT) and International Air Transport Association (IATA) regulations governing the shipment of hazardous materials and dangerous goods are followed. These regulations (49 CFR Parts 171 – 180 and Dangerous Goods Regulations For IATA) describe proper marking, labeling, packaging, and shipping of hazardous materials.

The definitions of dangerous goods and hazardous materials as described in DOT and IATA:

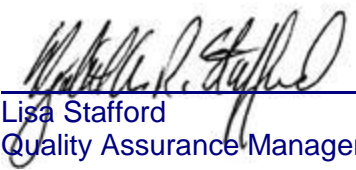

Dangerous Goods – “Articles or substances which are capable of posing a significant risk to health, safety, or to property when transported by air and which are classified according to the UN hazard classes.”

Appendix F

Sample Custody

Title: Sample Receipt and Procedures

[Quality Assurance Procedure]

Approvals (Signature/Date):			
	<u>05/10/2019</u>		<u>05/06/2019</u>
Kim Nelson	Date	Joe Schairer	Date
Technical Manager		Health & Safety Manager / Coordinator	
	<u>05/09/2019</u>		<u>05/08/2019</u>
Lisa Stafford	Date	Chris Williams	Date
Quality Assurance Manager		Laboratory Director	

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1. SCOPE AND APPLICATION

- 1.1. It is the responsibility of the employee to perform the procedure described here in full compliance with this SOP.
- 1.2. It is the responsibility of the Laboratory Director, QA Manager, and Departmental Supervisors of this facility to ensure that the analysis is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

2. SUMMARY OF METHOD

- 2.1. This SOP describes the procedures for laboratory chain-of-custody, including receipt and acceptance of sample shipments, storage requirements, generation of computer records, and corrective actions for sample receipt anomalies.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

4. INTERFERENCES

- 4.1. Any checks on samples or storage of samples should be done to eliminate any cross contamination.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

- 5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling closed sample containers.

- 5.1.2. Ice chests and shipping containers may be heavy. Always use safe lifting procedures. Whenever possible, use mechanical devices to lift or move containers from the floor to countertop/fume hood level to be unpacked.
- 5.1.3. Full ice chests larger than “lunchbox size” are not to be carried any further than ten feet. Instead, use a cart or scissors lift to move them. Repeated short trips can also present the same lifting/carrying hazard.
- 5.1.4. Any time a broken sample bottle is found inside an ice chest, continue to unpack the ice chest in the hood.
- 5.1.5. Associates opening any ice chests or shipping container must wear approved cut-resistant gloves while removing the samples. If any glass containers are found to be broken, cut resistant gloves must also be worn while discarding packing material and cleaning the shipping container. These can be Hyflex CR + (nitrile coated Kevlar) or MAPA Blue-Grip latex. Chemical protective gloves must be worn under or over these gloves, as needed. All packing materials are to be discarded in this situation.
- 5.1.6. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, sub-sampled and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.7. Laboratory procedures such as repetitive use of pipettes, repetitive subsampling, moving heavy shipping containers, unloading shipping containers, and manipulation of 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.
- 5.1.8. Safety policies apply to ALL sample administration visitors, including auditors, employees, couriers or clients who deliver samples.
- 5.1.9. Some types of biological samples may present special hazards. Refer to Appendix 8 of this document for more information.
- 5.1.10. Samples containing or potentially containing chemical warfare agents or degradates present a special hazard. Review Appendix 7 of this document before opening any coolers containing these types of samples.

5.1.11. Gloves will be changed immediately after handling AFFF product samples or samples with known gross contamination to prevent cross contamination.

Gloves need to be disposed of in the contaminated lab trash.

5.2. Primary Material 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
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrat or	1 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm- TWA 4 ppm- STEL	Nitric acid is extremely hazardous. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Bisulfate	Corrosive	None listed	Contact may cause skin/eye burns. Inhalation can cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing and shortness of breath. Causes chemical burns to the respiratory tract. May cause fatal spasms, inflammation or pulmonary/respiratory edema.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

- 6.1. Probe thermometer capable of reading to 0.1°C calibrated at a minimum of once annually against a NIST reference thermometer.
- 6.2. pH paper (Range pH 2 to pH 12 or equivalent).
- 6.3. Pipette.
- 6.4. Plain wood tongue depressors.

7. REAGENTS AND STANDARDS

- 7.1. Not applicable. All preservatives are applied in other departments. Associates must be aware of the hazards associated with those preservatives in Section 5.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. This SOP does not address sample collection.
- 8.2. Preservation and storage of samples is determined by each method. See method SOPs.

9. QUALITY CONTROL

- 9.1. Not applicable.

10. CALIBRATION

- 10.1. Thermometers are calibrated according to WS-QA-0016.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a non-conformance memo (NCM) and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The non-conformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A non-conformance memo shall be used for this documentation.

11.2. Sample Receiving Acceptance Policy

Before samples are received, the laboratory should provide the client's sample collection personnel the TestAmerica Sacramento Sample Receiving Acceptance Policy (see Appendix 1). It is sent along with the bottle order.

11.3. Receiving Shipment

For samples received "over the counter" rather than from a commercial carrier, see also section 11.14.

11.3.1. Shipments are received from a variety of commercial carriers. When shipments are received, record the time that the carrier arrived and the number of packages (including coolers) received on the daily shipment log. Refer to this time when entering the receipt time onto the Sample Receiving Notes form, and into the TALS system.

11.3.2. Verify accuracy of each shipping container's delivery address. Note the condition of the seal and if the custody seal number is present. Seals with evidence of tampering require that the client be notified and an NCM generated.

11.4. Opening the Shipment

11.4.1. Following safety policies, open the shipping container and remove paper work.

11.4.2. All shipping containers should be opened in a fume hood. After opening, the shipping container may be moved to a counter for further processing as long as it is determined that none of the following conditions apply. If any of the following conditions apply, the shipping container must remain under the fume hood during processing.

- The shipment is accompanied by MSD documentation,
- The container contains dry ice,
- Sample containers within the shipping container are broken or leaking,
- The shipping container exhibits a strong, noxious odor, or
- The shipment contains concentrated product samples.

11.4.3. If a shipment contains broken or leaking samples, notify the project manager via e-mail. Continue processing the shipment under the hood with cut-resistant gloves. Digital pictures of the broken samples may be taken and sent to the PM's e-mail upon request. If there are no intact samples for a given sample point and analysis, call the project manager so that the PM can notify the client as soon as possible. Dispose of broken samples according to the facility

hazardous waste procedures. The Hazardous Waste Specialist or Environmental Health and Safety Coordinator should be contacted if additional information is required.

- 11.4.4. AFFF product samples or samples with known gross contamination will be segregated from ultra trace PFAS samples and stored in R-22 to prevent possible cross-contamination.

11.5. Processing the shipment

- 11.5.1. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". Use the date and time that the cooler was received from the commercial carrier; this is recorded on the daily shipment log. This is also required for legal chain of custody. If the COC is not relinquished then file an NCM.
- 11.5.2. Record the tracking number from the air bill on the Sample Receiving Notes form and record whether or not the shipment came with a seal (if TestAmerica seal, record the red seal number). If the air bill indicates that the shipment is one out of several shipment containers, record this on the Cooler ID line on the Sample Receiving Notes form.
- 11.5.3. Immediately after opening, determine and record whether ice or artificial coolants are present. Measure the temperature and record the results on the Sample Receiving Notes form, noting both the observed and corrected sample temperatures. Record the corrected temperature on the COC as well. Make sure to fill out the rest of the Sample Receiving Notes form when necessary and initial and date the bottom. Note: If this process is interrupted at any point, a verbal hand-off to another technician must occur or the process must be restarted from the beginning.
 - 11.5.3.1. Use the AK thermometer by removing the thermometer from its casing, turning it on, and placing the thermometer probe into the temperature blank. If no temperature blank is provided, place the probe in between two of the sample containers, making sure to hold the containers together so that they are touching. If only one sample is received, place the sample against the interior cooler wall and place the probe in between the cooler wall and sample. Make sure to let the thermometer equilibrate before recording the temperature. Note any correction factors to be applied.
 - 11.5.3.2. If a random sample(s) is used to measure the temperature, make sure the sample(s) are placed back into the cooler until the cooler is being unpacked.

- 11.5.3.3. A non-conformance memo must be entered if the temperature reading is below 0°C or above 6°C. This notifies the project manager via email and is stored in TALS with the sample data. In the non-conformance memo software, be sure to include the client ID for all samples that are associated with a temperature exceedance.
- 11.5.3.4. Some sample matrices do not require cooling during transit. Situations where acceptable temperature range exceedance may be expected include, but are not limited to:
- Samples delivered within six hours of close of daily sampling event (Note: Cooling agent must be present).
 - Paperboard and dry pulp samples; non-conformance memo not necessary.
 - Dry incinerator ashes; non-conformance memo not necessary.
 - Samples for metals analysis only; non-conformance memo not necessary. Note: This does not apply to samples for mercury analysis. Such samples must be shipped on ice at 4 °C, and narrated with a non-conformance memo if not on ice.
 - Dry product samples; non-conformance memo not necessary.
 - Volatile air samples in bags or canisters.
- 11.5.4. Start a folder for the login. Use a manila folder unless the samples are part of a rush order, in which case use a red folder, or if the samples require subcontracted tests and will be sent elsewhere, in which case use a blue folder. Rush samples will usually be labeled with a “RUSH” sticker. Typically for subcontracted tests, the project manager will send out an e-mail before receiving the samples so that they can be caught or it will be clear based on the COC that the samples are for subcontracted tests.
- 11.5.5. Place the COC in the folder along with the Sample Receiving form.
- 11.5.5.1. Certain clients may require that the shipping manifest/air bill be included in the documentation; therefore, all seals, waybills and airbills will be saved. The list of clients who require this can be found on the corkboard in the back of the lab. If the shipping manifest/air bill is not in good shape, a photocopy is acceptable to include in the documentation. The shipping manifest is an integral part of the project documentation and is included with the finalized project when required by the client or regulatory agency.

- 11.5.5.2. Certain clients (such as those in Alaska) may require that the custody seals or a photocopy of the seals be kept for inclusion in the permanent record.
- 11.5.6. Examine accompanying documentation. If documentation is absent contact the lead project manager for assistance.
- 11.5.7. Unpack the samples. If the client/site requires association of samples to specific coolers, keep the samples separated based on the cooler in which they arrived, and label each group accordingly. Otherwise, arrange the samples by the order on the COC.
 - 11.5.7.1. Samples that are AFFF product samples will be kept separate from other samples on a separate sample cart while being checked in and until labeling begins. Fresh bench paper should be placed on the sample cart before samples are taken out of the cooler and disposed of in the contaminated lab trash. Frozen samples will be placed in a plastic bag as a secondary container and placed in freezer F10.
 - 11.5.7.2. If samples will be exposed to ambient temperatures for more than 20 minutes after they are removed from the cooler, the samples requiring temperature preservation should be placed in a sample storage refrigerator (volatile samples stored separately) until the sample login process can begin.
 - 11.5.7.3. If samples requiring thermal preservation (i.e., cold storage) exceed temperature requirements during login, the client must be notified in writing. File an NCM in TALS to notify the PM so that the PM can notify the client.
- 11.5.8. Dispose of packing materials appropriately based on the type of sample received. Packing materials for water samples are collected in uncontaminated lab trash cans. Packing materials from soil samples are collected in yellow lab trash cans prior to disposal in a steel waste drum. Packing materials from coolers containing broken sample jars or bottles, no matter what the type of sample, must be disposed of into an orange trash can or the steel waste drum in the H3 closet.
- 11.5.9. Use the bottle inventory (Appendix 3) as an aid when logging in samples. Examine any VOA vials for bubbles, and note the number of containers with bubbles on the bottle inventory. Compare the sample containers and their preservation to the list of analyses requested. Although some exceptions are permitted regarding certain clients, container types must compare to those listed in the Sample Receiving Reference Guide, CA-Q-WI-025SAC. Record any variations from the above expectations on the COC and file a non-

conformance memo.

11.5.10. If Terra Core sample kits are present in the shipment (detailed instructions available in form QA-385 Terra Core Instruction (Appendix 17)):

- The Terra Core kit consists of three 40 mL VOA vials, a 20 mL polyethylene bottle, and a 4 oz or 8 oz glass jar.

Note: The VOA vials may be in a plastic bag. DO NOT remove the vials from the bag.

- Proceed with labeling the 20 mL polyethylene and glass jars. DO NOT label the 40 mL VOA vials.

11.5.11. If Encore® sample containers are present in the shipment:

- Send e-mail notification to the distribution list: Sacramento-Rush GCMS and include the associated PM.
- Send the e-mail as priority. This will help the VOA department with prioritization.
- Use a subject line of “Encores to preserve” and the TALS job number.
- Include the date and time the first sample will expire. This will also assist the VOA department in prioritization. If the sample expires in less than 2 hours, notify all analysts in the department as well.
- Store the encores in “V” (R-14) fridge and post the red “ENCORES” sign on the fridge. This sign will alert the analyst that there are samples in the fridge for pickup. Once the samples have been picked up, the red sign on the fridge will be changed to a green colored “NO ENCORES”.

11.5.12. Based on workload, samples may now be stored with their folder, chain of custody, and complete inventory of samples received until the remaining login steps are performed.

11.6. Prioritizing Workload for Login

Frequently, the shipping containers from a given carrier will have samples from multiple clients with diverse needs. To ensure prompt and timely service while meeting the needs of the clients, it is necessary to prioritize the order in which sample deliveries are logged in.

11.6.1. Prioritize the shipment according to the Turn Around Time (TAT), while ensuring extraction holding times have not been exceeded. "RUSH" projects are placed in a red folder, projects requiring a project manager's immediate attention to add tests are placed in blue folders, and all other projects are placed in manila folders. The affected department is to be notified by email or page when the samples are available.

- 11.6.2. For each client/site, examine the analyses requested and the sampling dates/times. Be aware of any notation for expected TAT, or a due date that is less than 10 days, or hold times that will expire within 72 hours. Most aqueous organic analyses require extraction within seven days of sampling. If three days or less are remaining, consider the lot to be a priority and file in a manila folder (Method 8290 dioxin/furan analyses are an exception, requiring extraction within thirty days).
- 11.6.3. Any analysis that has a standard holding time of forty-eight hours or less is considered a "Short-Holding Time" (SHT) analysis. These are reported to the affected department immediately with the Short Holding Time Test notification (see Appendix 2) with a copy of the COC attached to the form. The receiving chemist initials and records the date and time on the SHT form. Any requested TAT of 10 days or less is also considered a priority.

11.7. Sample Login

- 11.7.1. Select the project with the highest priority:
- Short holds are processed immediately.
 - Blue folder (requires a project manager's immediate attention to add test and/or arrangements for sending samples to subcontract laboratories ("send outs"))
 - Red folder (rush)
 - Manila folder (normal TAT)
 - Grey folder (Air)
- 11.7.2. Line the samples up on a counter. Organize them by the order on the COC. If the client requires association of containers to coolers, make sure to keep that association when lining up the samples. Keep in mind that samples requiring thermal preservation should not be exposed to ambient temperatures for more than 20 minutes during the login process. Work on a single set of samples (i.e., by client and site) at a time.
- 11.7.3. Read shipment documentation. Ideally, samples are to have possession documented on a COC form. The COC will identify samples individually by alphanumeric designators, list sampling dates/times for each sample, requested analyses and document possession (via Relinquished by; and Received by; signatures with date and time of relinquishing/receiving). Signatures of possession qualify samples as having been "received under Chain-of-Custody". A Letter-of-Transmittal is also accepted as definitive documentation. Other forms of documentation include Request for Analysis, Shipping Order, Purchase Order, and various computer listings of sample

information. If no documentation that lists sample identifications exists, complete a COC when accepting the samples.

- 11.7.4. Check the samples against the COC for accuracy (e.g., sample ID, collection date/time, etc.). If the samples have not been received in good condition, it must be noted on the COC and an NCM must be filed. All discrepancies must be noted as well, including the lack of a relinquishing signature from the shipper.

Note: Good condition is loosely defined as all containers intact with no obvious discrepancies present. The shipment is estimated at this time to be viable; what is being requested coincides with what has been received. Temperature exceedances and minor discrepancies become issues when so specified by contract or client instruction. The presence of bubbles in volatile containers is documented. All such observations must be documented on custody chains, and by using the non-conformance memo software.

- 11.7.5. For all work sampled at or concerning government property, any federal projects, or as specified in client QAPPs, the pH of the preserved aqueous samples must be checked and recorded on Sacramento Sample Receiving Preservation Check Form QA-611 (Appendix 4). VOA vials are exceptions from this requirement. The pH is notated with a check mark or X per container type if it meets specifications. If the pH is out of specification, record the actual pH.
 - 11.7.5.1. After placing the samples in the hood, invert the sample container three times, remove the cap and pour a small aliquot of the sample into the lid. Pour the aliquot from the lid onto a fresh piece of pH indicator paper. Compare against the pH color grid table located on the pH strip container. Recheck readings that indicate samples were unpreserved when they should have been. Note discrepancies on the COC. Notify the project manager and file a non-conformance memo for the concerned sample.
 - 11.7.5.2. For metals samples that should be preserved but are received unpreserved, page the metals department. A technician will preserve the samples and document the preservation. An NCM must be filed for this anomaly.
 - 11.7.5.3. Store the completed preservation check form in the folder.
- 11.7.6. Check the walk-in fridge and other storage areas to determine the storage location(s) for the samples. Designate a storage location using the following guide:

- 11.7.6.1. R-14 (V) - volatile containers awaiting transfer to the VOA group refrigerators.
- 11.7.6.2. F10 - samples requiring freezing, including plant/animal tissues, and soil samples for method 1668 and 1699. "Samples stored in glass jars which are preserved by freezing must be placed in a Ziploc bag for secondary containment in case of glass breakage."
- 11.7.6.3. R22 - any samples where secondary containment in Ziploc bags is deemed necessary due to smells, spillage, degradation of container exterior or suspected high concentration of analytes.
- 11.7.6.4. C1 - dry pulp, paper samples or cassettes.
- 11.7.6.5. EPA1 - soil samples received from the Environmental Protection Agency, solvents, air toxics projects for method 29, or clients requiring dioxin work under SOW DFLM01.1.
- 11.7.6.6. Walk-In – Main sample storage area. If samples do not meet any of the above requirements, they may be stored in the Walk-In cooler.

Note: Location selections specify shelf by letter, example: W4A.

Note: Dioxin soil samples received in clear glass jars must be stored in boxes to protect from light.

- 11.7.7. Open TALS. Enter the Sample Management tab. Navigate to the login tab, and use information from the COC and the project manager to obtain a project number to associate with the login. Enter the login number on the top section of the bottle lot inventory and record on the COC.
 - 11.7.7.1. Enter information into the Receipt/Info and Receipt/Containers tabs. If the client requires association of containers to coolers, be sure to enter the specific cooler for each container in the cooler field for the container.
 - 11.7.7.2. Complete the receipt/checklist tab. Complete anomalies if prompted.
 - 11.7.7.3. Complete the information on the Login/Samples and Login/Samples tabs.
 - 11.7.7.4. File any holding-time violation reports (HTV), if necessary, by clicking the "NCM" button, and entering the appropriate information. Answer the queries, and be certain to mail the HTV to the department supervisor and project manager involved. The HTV

is filed by sample administration when a holding time is expired upon receipt. If the holding times have not completely expired for a project, the department handling the analysis in question files the HTV.

- 11.7.7.5. When all information has been entered, print the sample labels.
- 11.7.8. Label sample containers. Whenever possible teams of two people should be labelling, with one person verifying client ID on bottle and the bottle type against the TALS label prior to applying the label, and the second to check the information on the labelled bottles. Be certain the labels adhere to containers. When possible, place the large labels (Appendix 5.3a) along the long axis of the bottle, so that the barcode is not wrapping around a curve. Unless the container is a VOA vial, place the small label (Appendix 5.3b) on the container lid. Attempt to leave all client label information exposed. When this is not feasible, affix label so that the client ID, sampling date/time, and preservative are showing. Preprinted laboratory names may be covered with no consequence. If label adhesive is insufficient, use cellophane tape to secure label. Place sample in Ziploc bag when storage location is R22. Labeling may be peer-reviewed whenever the complexity of the project warrants a second check to ensure accuracy. A peer compares the order of the samples to the documentation and ensures that all containers of a sample have the same sample number and are sequential. When placing samples on the storage shelves, verify that the bottle location identified on the label matches the location samples are being placed.
- 11.7.8.1. Circumstances where a review is warranted may include:
- A large number of samples are present.
 - Many containers per sample are present.
 - VOA containers are present.
 - Client identifications are illegible or confusing.
 - A review is requested by a TestAmerica Sacramento employee.
 - When training new personnel
 - When the process of pairing off cannot be followed.
- 11.7.8.2. Fresh bench paper must be laid on the counter prior to lining up and labelling any AFFF product samples or samples with known gross contamination to prevent cross contamination. Also, bench paper must be disposed of in the contaminated lab trash can immediately following AFFF product sample labelling.
- 11.7.9. Scan the COC and attach to the job in TALS. Scan any additional

documentation (listed in 11.7.10 below) as “field sheets” and attach to the job in TALS.

11.7.10. When login is complete, place the folder in the project manager's tray in sample administration. At this time, the folder should contain:

- Original COC.
- Air bill and secondary documentation, if present.
- Bottle lot inventory.
- Sample Receiving Notes form.
- Preservation check form, if preservations checks performed.

11.8. Shipping Container Return

11.8.1. Broken samples and packing material contaminated with spilled samples **MUST** be disposed of as hazardous lab trash. Shipping containers must be decontaminated before being put back into use. Decontamination procedures will depend on what was spilled. See EH&S staff for specific instructions.

11.8.2. Except when discarded, shipping containers are cleaned in bottle prep or returned to the client. Containers belonging to TestAmerica Sacramento are marked with a permanent marker. If the container belongs to a client and they request a return, either a return label will be included in the shipment or the client will make a note requesting a return. Make sure that the return address is saved in this case. All hazardous materials labeling must **be removed or defaced** in some way. Dry the interior of the container if wet, replace packing and return to bottle prep.

11.8.3. Packing and artificial ice that is deemed reusable may be returned to the client. Packing that resembles trash or is ruined during unpacking is disposed as trash. Drain any water from the container before sealing it closed. Due to partially frozen artificial ice, client containers may not be perfectly dry when returned. Complete a return-mailing label if necessary. Secure it with cellophane tape and seal the container.

11.8.4. Temperature blanks are kept and reused.

11.9. Per USDA permit regulations, ice chests that have been used to ship soil samples must be decontaminated with 10% bleach solution during the cleaning process before being re-used or shipped empty to another location.

11.9.1. Ice chests that have had soil samples are marked with a label that reads, ”Soil Sample – Clean with Bleach” and moved to the bottle prep area where they are cleaned with 10% bleach.

11.10. Disposal of Ice

11.10.1. When ice is present, it is often enclosed in plastic bags. A basin used for dumping ice is located in sample administration. Open the bags and dump ice into the basin. Water should not be left running in the basin as the basin drains into a closed system. When excessive amounts of ice are received, collect the ice in an ice chest and dump it over the storm drains outside the building.

This is permissible only if the ice and coolers are uncontaminated.

Additionally, ice used to ship any soil samples may not be disposed down the storm drain. It may only be disposed of down the basin in sample administration.

11.11. Refrigerator and Freezer Temperatures

11.11.1. Refrigerator and freezer temperatures will be monitored and recorded once daily Monday through Saturday. Any temperature below 0°C or above 6°C for the refrigerators and above -10°C for the freezer will need corrective action. Refer to WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers. Temperatures will be recorded on the appropriate charts. Temperature charts will then be stored in the temperature logbook. All charts for a particular refrigeration unit will be stored together in chronological order.

11.12. Internal Sample Tracking

Samples are tracked within the facility using the Internal Chain of Custody (ICOC) feature of TALS.

11.12.1. In TALS, the analyst selects Internal Chain of Custody and ascertains storage location. Then the analyst removes appropriate containers from storage unit and organizes them by lot and sample number. Analyst completes checkout portion of internal COC in TALS using the barcode scanner.

11.12.2. If a sample is completely used up in the extraction laboratory, then the disposition of the container must be documented. All empty containers not returned to sample administration must be recorded as destroyed in testing on the ICOC. If the containers are not to be returned and will enter the waste stream through the extraction lab, the analyst will also enter this information during checkout.

11.12.3. Analyst completes the checkout process before leaving sample control. At return, the analyst completes the check-in portion of the ICOC. Analyst returns samples to their storage location. If subsampling or aliquoting is performed, whether outside the area or not, containers must be checked out by the analyst. The original containers' whereabouts and those of any other

containers generated must be documented.

11.13. Sending Out Subcontracted Work

- 11.13.1. In the event samples require being subcontracted to another lab, the samples will be processed in the very same manner as all other samples. Samples should be received and processed as described in Sections 11.1 to 11.7 of this SOP. The project manager should notify the sample receiving staff as to all samples they require to be shipped out.
- 11.13.2. Using TALS, generate a new TestAmerica Sacramento chain of custody for the subcontracted samples by scanning out the containers to the desired lab. Contact the project manager for assistance if any difficulties are encountered. Project Managers must verify samples to be sent out and relinquish the COC.
- 11.13.3. Samples should be packaged in a manner in accordance with the TestAmerica Sacramento Receiving Acceptance Policy. Additional packing may be used to ensure samples have adequate protection from breakage. Samples are subsequently packaged in a bag containing copious amounts of uncontaminated wet ice. There should be sufficient ice to maintain the samples within the temperature range of 0°C to 6°C throughout transit. The ice packed samples are stored in the appropriate sized insulated container.
- 11.13.4. Ensure the COC is packed in the shipping container and that it is protected from moisture from ice and/or samples (in the event any containers become compromised).
- 11.13.5. The packed container is then sealed with a signed and dated Custody Seal and a generous amount of shipping tape to ensure the contents remain secured. Ensure they are labeled with the appropriate shipping labels and delivered to the courier. Add the tracking number to TALS.

11.14. Client Deliveries ("Over the Counter")

- 11.14.1. Samples received “over the counter” are samples hand delivered by the client, sampler or courier directly employed by the client's company. The pivotal question in such deliveries is whether the person possessing the shipment is in custodial possession of the samples or merely transporting the shipment. If in custodial possession, the deliverer will need to relinquish custody to you before departing and will probably retain a copy of the documentation. To determine the matter, simply ask the deliverer if they have signed any of the sample documents.
- 11.14.2. Mention any discrepancies and allow corrections to be made by the client,

sampler or courier. Also ask for any special instructions or important aspects of the project (i.e. rush turn-around times, impending hold-time violations, sample matrix specifics, etc.). Solicit the client's needs such as shipping containers, bottle orders, to speak with a project manager or any other requests. After the client's departure, complete any aspects of Sections 11.1 through 11.15 left unfinished. Prioritize the new project within the existing workload and proceed with the highest priority.

11.15. For after hours sample receipt instructions for non-sample administrative personnel, refer to Appendix 9.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. The Department Manager 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.

14. POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.1. All ice, or melted ice, that has been used to store or ship any soil samples, or in any container with soil samples, must be allowed to melt through a 100 mesh screen, in order to comply with our USDA soil permit.

14.2. Per USDA permit regulations, ice chests that have been used to ship soil samples must be decontaminated with 10% bleach solution during the cleaning process before being re-used or shipped empty to another location.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste

disposal procedures are incorporated by reference to WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Uncontaminated packing materials such as vermiculite, bubble wrap, plastic bags, paperwork, etc. These are collected in the uncontaminated lab trash cans and are disposed of in the dumpster at the end of the day.
- 15.2. Packing materials containing soil samples or contaminated solid packing materials, including broken glass, caused by the breakage of sample containers during shipment. Dump the solid waste into an orange contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the applicable 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. Contaminated melted ice and aqueous samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.
- 15.4. Contaminated melted ice and solid or soil samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.

16. REFERENCES/CROSS REFERENCES

- 16.1. Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth edition, January 2005, Appendix A.
- 16.2. Sacramento Glass Safety SOP, WS-QA-0025, current revision.

17. METHOD MODIFICATIONS

- 17.1. There are no deviations from the method.

18. ATTACHMENTS

- 18.1. Appendix 1 – Eurofins TestAmerica Sacramento Sample Receiving Acceptance Policy
- 18.2. Appendix 2 – QA-356 Short Hold Test Notification
- 18.3. Appendix 3 – QA-185 Bottle Lot Inventory
- 18.4. Appendix 4 – QA-611 Sample Receiving Preservation Check

- 18.5. Appendix 5 – Example Labels
- 18.6. Appendix 6 – Sample Receipt Flow Chart
- 18.7. Appendix 7 – Chemical Warfare Degradates – Potential Hazards in Sample Receipt
- 18.8. Appendix 8 – Handling of Blood or Other Potentially Infectious Materials
- 18.9. Appendix 9 – PFAS Bottle Prep and Sample Handling
- 18.10. Appendix 10 – Sample Receipt and Procedures Training Guide
- 18.11. Appendix 11 – TALS Checklist Training Guide
- 18.12. Appendix 12 – After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel
- 18.13. Appendix 13 – QA-812 Sample Receipt Notes
- 18.14. Appendix 14 – QA-828 Canister Receipt Notes
- 18.15. Appendix 15 – CA-Q-WI-031 Revenue Source Tracking Procedure
- 18.16. Appendix 16 – QA-836 Sample Receipt and Procedures Checklist
- 18.17. Appendix 17 – QA-835 Terra Cores Instruction

19. REVISION HISTORY

- 19.1. WS-QA-0003 Revision 12.7, Effective 05/15/2019
 - 19.1.1. Section 11.4.3 revised, “Digital pictures may be taken of the broken samples at the and the files attached to the project manager’s e-mail” to “Digital pictures of the broken samples may be taken and sent to the PM’s e-mail upon request.”
 - 19.1.2. Section 11.5.3.1 added, “If only one sample is received, place the sample against the interior cooler wall and place the probe in between the cooler wall and sample.”
 - 19.1.3. Section 11.7.8 added, “When placing samples on the storage shelves, verify that the bottle location identified on the label matches the location samples are being placed.”
 - 19.1.4. Section 11.7.9 added, “Scan any additional documentation (listed in 11.7.10

below) as “field sheets” and attach to the job in TALS.”

- 19.1.5. Section 11.7.10 revised, “temp record form” to “Sample Receiving Notes form” and added, “Preservation check form, if preservations checks performed.”
 - 19.1.6. Updated Appendix 1 with current version of, “Eurofins TestAmerica Sacramento Sample Receiving Acceptance Policy”.
 - 19.1.7. Updated Appendix 2 with current version of, “QA-356 Short Hold Test Notification”
 - 19.1.8. Updated Appendix 3 with current version of, “QA-185 Bottle Lot Inventory”.
 - 19.1.9. Updated Appendix 4 with current version of, “QA-611 Sample Receiving Preservation Check”.
 - 19.1.10. Updated Appendix 13 with current version of, “QA-812 Sample Receipt Notes”.
 - 19.1.11. Updated Appendix 14 with current version of, “QA-828 Canister Receipt Notes”.
 - 19.1.12. Updated Appendix 15 with current version of, “CA-Q-WI-031 Revenue Source Tracking Procedure”.
 - 19.1.13. Updated Appendix 16 with current version of, “QA-836 Sample Receipt and Procedures Checklist”.
 - 19.1.14. Updated Appendix 17 with current version of, “QA-835 Terra Cores Instruction”.
 - 19.1.15. Editorial changes.
- 19.2. WS-QA-0003, Revision 12.6, Effective 01/16/2018
- 19.2.1. Section 11.5.3 added note, “If this process is interrupted at any point, a verbal hand-off to another technician must occur or the process must be restarted from the beginning.”
 - 19.2.2. Section 11.7.2 added, “Work on a single set of samples (i.e., by client and site) at a time.”
 - 19.2.3. Section 11.7.8 added, “Whenever possible teams of two people should be labelling, with one person verifying client ID on bottle and the bottle type

against the TALS label prior to applying the label, and the second to check the information on the labelled bottles.”

- 19.2.4. Section 11.7.8.1 added, “when training new personnel”, and “When the process of pairing off cannot be followed.”
- 19.2.5. Updated Appendices 13 and 14 with current form versions.
- 19.2.6. Editorial changes.
- 19.3. WS-QA-0003, Revision 12.5, Effective 05/15/2018
 - 19.3.1. Section 5.1.4 revised to, “Any time a broken sample bottle is found inside an ice chest, continue to unpack the ice chest in the hood.”
 - 19.3.2. Removed Section 6.1, “IR thermometer calibrated at a minimum of once per quarter against an NIST reference thermometer.”
 - 19.3.3. Removed Section 6.2, “Filament thermometer calibrated at a minimum of once annually against an NIST reference thermometer.”
 - 19.3.4. Added Section 6.4, “Plain wood tongue depressors.”
 - 19.3.5. Removed Section 10.2, “All electronically operated thermometers (including IR thermometers) must have their calibration verified each day of use. IR thermometers will be checked against mercury thermometer ID 4881 and the corrected temperature must be within 1 degree. If the corrected temperature is greater than 1 degree then QA must be notified and that thermometer will not be used until it is recalibrated. IR thermometers are to be calibrated quarterly against a NIST reference thermometer.”
 - 19.3.6. Removed Section 11.5.3.1, “Use the infrared thermometer by directing the thermometer at the temperature blank container (or a random sample container if no temperature blank is provided), making sure that no labels or packaging materials are interfering with the direct contact of the infrared beam and the container.”
 - 19.3.7. First sentence of Section 11.5.5.1 changed to, “Certain clients may require that the shipping manifest/air bill be included in the documentation; therefore, all seals, waybills and airbills will be saved.”
 - 19.3.8. Section 11.5.7.1 revised to, “Samples that are AFFF product samples will be kept separate from other samples on a separate sample cart while being checked in and until labeling begins. Fresh bench paper should be placed on the sample cart before samples are taken out of the cooler and disposed of in

the contaminated lab trash. Frozen samples will be placed in a plastic bag as a secondary container and placed in freezer F10.”

- 19.3.9. Section 11.5.9 revised to, “Use the bottle inventory (Appendix 3) as an aid when logging in samples. Examine any VOA vials for bubbles, and note the number of containers with bubbles on the bottle inventory. Compare the sample containers and their preservation to the list of analyses requested. Although some exceptions are permitted regarding certain clients, container types must compare to those listed in the Sample Receiving Reference Guide, CA-Q-WI-025SAC. Record any variations from the above expectations on the COC and file a non-conformance memo.”
- 19.3.10. Added Section 11.5.10, “If Terra Core sample kits are present in the shipment (detailed instructions available in form QA-385 Terra Core Instruction):”, and its associated subsections detailing the handling of Terra Core kits.
- 19.3.11. Section 11.6.3 inserted, “with a copy of the COC attached to the form.”
- 19.3.12. Section 11.7.3 inserted, “(via Relinquished by; and Received by; signatures with date and time of relinquishing/receiving).”
- 19.3.13. Added Appendix 16, “Sample Receipt and Procedures Checklist Example”.
- 19.3.14. Added Appendix 17, “Terra Cores Instruction Example”.
- 19.3.15. Throughout SOP updated references of “temporary record form” to “Sample Receiving Notes form”.
- 19.3.16. Editorial changes.
- 19.4. WS-QA-0003, Revision 12.4, Effective 10/31/2017
 - 19.4.1. The revision history prior to 2016 has been removed. It may be viewed in earlier versions of this SOP.
 - 19.4.2. Added Section 5.1.11, “Gloves will be changed immediately after handling AFFF product samples or samples with known gross contamination to prevent cross contamination. Gloves need to be disposed of in the contaminated lab trash.”
 - 19.4.3. Added Section 11.5.7.1, “Samples that are AFFF product samples will be kept separate from other samples on a sample cart while being checked in. Fresh bench paper should be placed on the sample cart before samples are taken out of the cooler and disposed of in the contaminated lab trash.”

- 19.4.4. Added Section 11.5.7.3, “If samples requiring thermal preservation (i.e., cold storage) exceed temperature requirements, during login, the client must be notified in writing. File an NCM in TALS to notify the PM so that the PM can notify the client.”
- 19.4.5. Added Section 11.5.8, “Dispose of packing materials appropriately based on the type of sample received. Packing materials for water samples are collected in uncontaminated lab trash cans. Packing materials from soil samples are collected in contaminated lab trash cans prior to disposal in a steel waste drum. Packing materials from coolers containing broken sample jars or bottles, no matter what the type of sample, must be disposed of directly into the steel waste drum.”
- 19.4.6. Section 11.12.3, removed “Transfer of volatile containers from VOA to RD and RF do not require verification.”
- 19.4.7. Section 15.2 added, “Packing materials containing soil samples or...”
- 19.4.8. Added Section 11.4.4, “AFFF product samples or samples with known gross contamination will be segregated from ultra trace PFAS samples and stored in R-22 to prevent possible cross-contamination.”
- 19.4.9. Added Section 11.7.8.2, “Fresh bench paper must be laid on the counter prior to lining up and labelling any AFFF product samples or samples with known gross contamination to prevent cross contamination. Also, bench paper must be disposed of in the contaminated lab trash can immediately following AFFF product sample labelling.”
- 19.4.10. Added Appendix 9, “PFAS Bottle Prep and Sample Handling”.
- 19.4.11. Added Appendix 10, “Sample Receipt and Procedures Training Guide”.
- 19.4.12. Added Appendix 11, “TALS Checklist Training Guide”.
- 19.4.13. Editorial changes.
- 19.5. WS-QA-0003, Revision 12.3, Effective 03/22/2017
 - 19.5.1. Section 11.4.2, removed “Under the following conditions, the shipping container must be opened in a fume hood, or, if the condition is discovered upon opening, it must be moved to a fume hood before further processing.
 - 19.5.2. Section 11.4.2, added “All shipping containers should be opened in a fume hood. After opening, the shipping container may be moved to a counter for further processing as long as it is determined that none of the following

conditions apply. If any of the following conditions apply, the shipping container must remain under the fume hood during processing.”

- 19.5.3. Section 11.4.3, removed “The client program requires opening within a fume hood.”
- 19.5.4. Section 11.5, split up into sections.
- 19.5.5. Section 11.5.1, moved “If the COC is not relinquished then file an NCM,” into this section from Section 11.5.3.
- 19.5.6. Added Section 11.5.2, “Record the tracking number from the air bill on the temporary record sheet and record whether or not the shipment came with a seal (if TestAmerica seal, record the red seal number). If the air bill indicates that the shipment is one out of several shipment containers, record this on the Cooler ID line on the temporary record sheet.”
- 19.5.7. Section 11.5.3, removed “...ascertain if ice or artificial coolants are present. Use the date and time received by the commercial carrier. If the COC is not relinquished then file an NCM. Measure temperature and record the results on the temporary record form.”
- 19.5.8. Section 11.5.3, added “...determine and record whether ice or artificial coolants are present. Measure the temperature and record the results on the temporary record form, noting both the observed and corrected sample temperatures. Record the corrected temperature on the COC as well. Make sure to fill out the rest of the temporary record form when necessary and initial and date the bottom.”
- 19.5.9. Section 11.5.3.1, in place of “sample containers” used “the temperature blank container (or a random sample container if no temperature blank is provided).”
- 19.5.10. Section 11.5.3.1, removed “Note the uncorrected and corrected sample temperature on the sample receiving notes page and write the corrected temperature on the COC. If the COC lists a temperature blank, or one is present in the shipping container, locate the blank and measure the temperature. If a temperature blank is not present, take the temperature of a random sample in the cooler to determine the temperature.”
- 19.5.11. Added Section 11.5.3.2 to read, “Use the AK thermometer by removing the thermometer from its casing, turning it on, and placing the thermometer probe into the temperature blank. If no temperature blank is provided, place the probe in between two of the sample containers, making sure to hold the containers together so that they are touching. Make sure to let the thermometer

equilibrate before recording the temperature. Note any correction factors to be applied.”

- 19.5.12. Added Section 11.5.3.3 to read “If a random sample(s) is used to measure the temperature, make sure the sample(s) are placed back into the cooler until the cooler is being unpacked.”
- 19.5.13. Section 11.5.3.5, added “Volatile air samples in bags or canisters.”
- 19.5.14. Section 11.5.4, added “Use a manila folder unless the samples are part of a rush order, in which case use a red folder, or if the samples require subcontracted tests and will be sent elsewhere, in which case use a blue folder. Rush samples will usually be labeled with a “RUSH” sticker. Typically for subcontracted tests, the project manager will send out an e-mail before receiving the samples so that they can be caught or it will be clear based on the COC that the samples are for subcontracted tests.”
- 19.5.15. Section 11.5.5, removed “Photocopy the shipping manifest/airbill and add to the sample receiving notes page and include the 8 ½” x 11” facsimile in the folder.
- 19.5.16. Added Section 11.5.5.1 to read “Certain clients may require that the shipping manifest/air bill be included in the documentation. The list of clients who require this can be found on the corkboard in the back of the lab. If the shipping manifest/air bill is not in good shape, a photocopy is acceptable to include in the documentation.”
- 19.5.17. Added Section 11.5.7.1 to read “If samples will be exposed to ambient temperatures for more than 20 minutes after they are removed from the cooler, the samples requiring temperature preservation should be placed in a sample storage refrigerator (volatile samples stored separately) until the sample login process can begin.”
- 19.5.18. Section 11.7.2, added “Keep in mind that samples requiring thermal preservation should not be exposed to ambient temperatures for more than 20 minutes during the login process.”
- 19.5.19. Section 11.5.8, changed “TestAmerica Sacramento Quality Assurance Manual Tables 22.1 through 22.8” to “Sample Receiving Reference Guide, CA-Q-WI-025SAC.”
- 19.5.20. Section 11.10.1, removed “...and the amounts disposed in this way are recorded.”

- 19.5.21. Appendix 1, updated Sample Acceptance Policy document to latest version (02/02/2015)
- 19.5.22. Appendix 10, updated Sample Receiving Notes document to latest version (Rev. 1.3)
- 19.5.23. Editorial changes.
- 19.6. WS-QA-0003, Revisions 12.2, Effective 11/11/2016
 - 19.6.1. Section 11.4.4.2 – Replaced LIMS with TALS.
 - 19.6.2. Section 11.7.8 – Replaced ‘login function’ with ‘Sample Management tab’.
 - 19.6.3. Updated Appendix 2 and Appendix 11 forms.
 - 19.6.4. Editorial changes.
- 19.7. WS-QA-0003, Revision 12.1, Effective 04/11/2016
 - 19.7.1. Edited paragraphs 5.1.2 and 5.1.5 to reflect practices on opening sample shipping containers and cleaning containers that are found to have broken glass sample containers inside of them.
 - 19.7.2. Added paragraph 5.1.3 regarding lifting and movement of full ice chests.
 - 19.7.3. Edited paragraphs 11.8 and 14.2 regarding cleaning of ice chests that had soil samples in them.
 - 19.7.4. Added paragraph 16.2, Glass Safety SOP.
 - 19.7.5. Editorial changes

APPENDIX 1

Sample Acceptance Policy

Environment Testing
TestAmericaSACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY*(Effective 05/15/2019)*

Page 1 of 2

The TNI Standard and Eurofins TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. Eurofins TestAmerica Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

VOA vials should be stored in controlled conditions. Exposure of trip blanks to temperature fluctuations is likely to cause development of bubbles in the trip blanks.

When completing the chain of custody form, please note that you must sign your name in the "relinquished by" box.

Requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking water samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- Containers of water meant for perchlorate analysis should have adequate headspace to prevent anaerobic microbial degradation. A void approximately 1/3 of the container volume is sufficient.
- The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0° C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.
 1. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements above. In these cases, the samples shall be considered acceptable if the samples were received on ice.
 2. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
 3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.
- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.


Environment Testing
TestAmericaSACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY*(Effective 05/15/2019)*

Page 2 of 2

- For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).
- The client must inform the laboratory if the samples are suspected to contain high levels of target analytes (>1 ppm levels for organics: >1ppb levels for dioxins, PCB congeners and PFAS). **Unannounced** samples grossly contaminated with dioxins, PCB congeners or PFAS (>1ppb) can have a severe impact on the laboratory's glassware and instrumentation. It is important our clients share in this risk and be held financially accountable for destroyed glassware that cannot be cleaned to trace level requirements.
- For AFFF samples, due to the potential for high concentrations of PFASs, it is critical that the associated chain of custody and sample containers clearly state that they are AFFF related samples.
- Air canisters (SUMMA® and other brands) have additional requirements:
 - Never write or affix a label directly on a canister. A special tag is attached to each canister for this purpose.
 - Complete the Canister Field Data Record with the initial and final vacuum/pressure reading for each canister during sampling.
 - Close all valves completely prior to shipping or transporting.
 - Return canisters, filters, flow controllers, vacuum flow regulators, and any other supplied equipment must be returned even if they were not used. Pack equipment carefully to minimize in-transit damage. Sampling equipment that is damaged, lost or not returned will be invoiced to the client at the replacement cost. Delayed return of equipment to the laboratory may result in additional rental charges.
 - Do not attempt to adjust or alter any equipment, as it may result in loss of sample integrity as well as equipment damage that may be invoiced to the client.

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

APPENDIX 2
Short Holding Test Notification

 <p>Environment Testing TestAmerica</p>	<p>West Sacramento SHORT HOLDING TEST NOTIFICATION</p>	
Client _____ Lot ID _____ Date & time received by Sample Control _____ Date & time received in Gen Chem _____	<input type="checkbox"/> SHT Analysis <input type="checkbox"/> Pending HTV (Routine Tests) <input type="checkbox"/> RUSH (24 - 48 - 72 HR TAT) <input type="checkbox"/> See Attached	
Chemist Notified _____ TestAmerica _____ _____ Holding time already violated? <input type="checkbox"/> Yes <input type="checkbox"/> No		
If HTV, has NCM been filed? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> MS/MSD required on sample # _____		
General Analysis (select those that apply)	Sample #	Expiration Date & Time
<input type="checkbox"/> pH <input type="checkbox"/> Alkalinity <input type="checkbox"/> Cr6+ <input type="checkbox"/> EC <input type="checkbox"/> pH-Soils <input type="checkbox"/> TDS/TSS	_____	_____
Method 353.2 <input type="checkbox"/> NO ₂ +NO ₃ <input type="checkbox"/> NO ₂ <input type="checkbox"/> NO ₃ ppm	_____	_____
Method 300.0 <input type="checkbox"/> NO ₃ <input type="checkbox"/> NO ₂ <input type="checkbox"/> OPO ₄ <input type="checkbox"/> Br <input type="checkbox"/> Cl <input type="checkbox"/> SO ₄ <input type="checkbox"/> F	_____	_____
<input type="checkbox"/> Other _____ _____ _____	_____	_____
<input type="checkbox"/> Chain of Custody attached		
Comments: _____ _____ _____		
<p style="font-size: small;">Q:\FORMS\QA-356 Short Holding Times QA-356 BWD 05/06/2019</p>		

APPENDIX 3
Bottle Lot Inventory

eurofins	Environment Testing TestAmerica		Bottle Lot Inventory																		
			Job ID: _____															Location: _____		Log #: _____	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	
VOA*	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	
VOAh*	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	/	
VOAmeoh																					
AGB																					
AGBs																					
250AGB																					
250AGBs																					
250AGBn																					
500AGB																					
___AGJ																					
500AGJ																					
250AGJ																					
125AGJ																					
125AGJmeoh																					
___CGJ																					
500CGJ																					
250CGJ																					
125CGJ																					
PJ																					
PJn																					
500PJ																					
500PJn																					
500PJna																					
500PJzn/na																					
250PJ																					
250PJn																					
250PJna																					
250PJzn/na																					
Acetate Tube																					
___CT																					
Encore																					
Folder/filter																					
PUF																					
Petri/Filter																					
XAD Trap																					
Ziploc																					
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	

h = hydrochloric acid s = sulfuric acid na = sodium hydroxide n = nitric acid zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

Q:\FORMS\QA-185 Bottle Lot Inventory.doc QA-185 BWD 05/06/2019

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APPENDIX 4

Sample Receiving Preservation Check


Eurofins TestAmerica		Sacramento Sample Receiving Preservation Check																											
Instructions: Check or x the container type, then check or x the pH measurement if it meets. Write the actual pH if out. Lot ID: _____		Container	250Pjn	500Pjn	BS	250AG	AGBh	AGBs	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	
		pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	pH ±12	
500Pj	na	500Pj	na	500Pj	na	500Pj	na																						
250Pj	na	250Pj	na	250Pj	na	250Pj	na																						
250Pj	na	250Pj	na	250Pj	na	250Pj	na																						
500Pj	zn/na	500Pj	zn/na	500Pj	zn/na	500Pj	zn/na																						
250Pj	na	250Pj	na	250Pj	na	250Pj	na																						

QA-611
BWD 05/03/2019

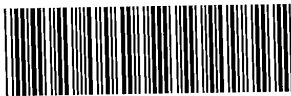
QA-611 Pres Check Sheet1

APPENDIX 5 Example Labels

1. Label for the Folder


	Loc: 320
Login: 320-1197	1197
PM: Sadler, Jeremy Company: TestAmerica Laboratories, Inc.	

2. Label for the COC

	Loc: 320
320-1197 Chain of	1197

3. Labels for the Samples



a. For the side of the container


320-1197-A-1
0114301X/51122/112812
Location: R14 shelf A VOA storage in Sample Contro
Bottle: Voa Vial 40ml - Hydrochloric Acid
Sampled: 11/28/2012 12:00 AM 320-47099 COC

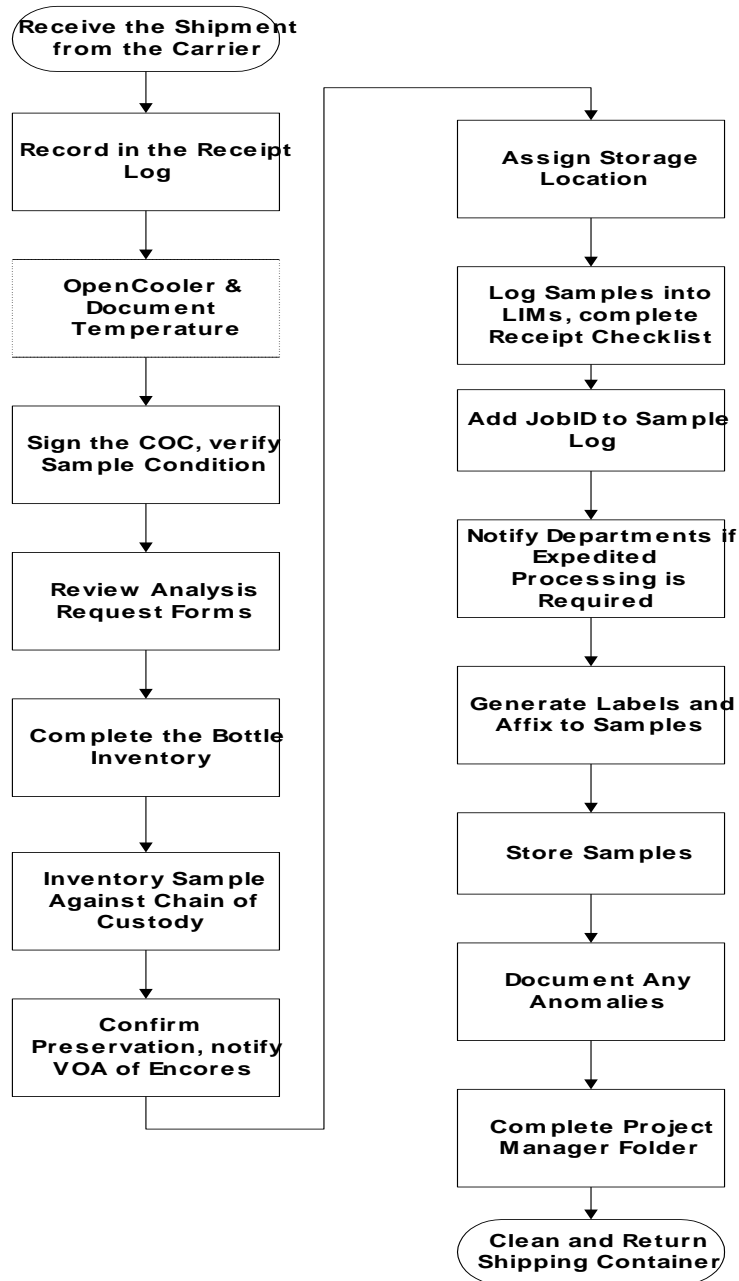
b. For the lid of the container

Loc: 320
1197
#1
A

4. Label for archive paperwork

	
550-61072 Field Sheet	550-61072 Field

**APPENDIX 6
Flow Diagram**



APPENDIX 7

Chemical Warfare Degradates - Potential Hazards in Sample Receipt

Background

TestAmerica Sacramento regularly receives samples to be analyzed for degradates of chemical warfare agents. These degradates generally are no more toxic than most of the compounds we deal with every day. The fact that these compounds are degradates of chemical warfare agents does, however, present a different type of potential hazard for us. We have developed policies regarding the handling of such samples. The purpose of this document is to discuss these compounds and the potential hazards involved in handling them.

Please note that TestAmerica Sacramento does NOT analyze samples for actual chemical warfare agents such as mustard, lewisite, Sarin, GD, VX, phosgene and tear gas. Parent compounds are analyzed by laboratories that have specialized personnel training, security, and handling procedures.

The toxicity of the by-products of some chemical warfare materials are more than the parent compound. An example of this is VX and EA 2192. Testing protocol for degradates is not necessarily specific for the analyte being screened for and the scientific community has not come to consensus on what “positive” test results actually mean. For this reason, in all cases where positive screening data is received for the parent compounds, Corporate EH&S, the Project Manager, local EH&S staff and senior management must be consulted before deciding to accept and proceed with handling such samples.

Review of Agent Compounds

Chemical warfare agents fall into a wide variety of categories, ranging from relatively mild chemicals such as tear gas to lethal nerve agents such as Sarin. The two types of agents we are most concerned with are **nerve agents** and **blistering agents**. Other lethal agents have been developed and tested for use in chemical warfare; however, these other compounds are either extremely volatile or reactive (and therefore highly unlikely to be present in an environmental sample) or were never produced in significant quantities in the U.S.

Nerve agents

These are members of the organophosphate class of compounds. They are similar to many common household pesticides such as diazinon. The difference is that nerve agents are far more toxic to humans. The first nerve agent, Tabun, was discovered prior to World War I by a German pesticide company during the process of screening new compounds for use as pesticides. Once the toxicity of Tabun was determined, various governments began screening many related compounds. Out of this effort have come five established (non-classified, i.e. public domain) agents. --- **Tabun (or GA), Sarin (or GB), GD, GF, and VX.**

All of these compounds are toxic via inhalation, ingestion, skin contact, or just about any other route of entry into the body.

A characteristic of these compounds is their volatility. A more volatile nerve agent will disperse

in air more effectively than a less volatile one.

Less volatile agents, on the other hand, will remain around on soil, vegetation, clothing, etc. and will therefore last longer. Out of these various agents, the only two produced in significant quantities in the U.S. are Sarin and VX. **Sarin is the most volatile** of the above agents while VX is the least volatile. Both are extremely toxic - a drop of pure VX barely visible to the naked eye is enough to kill a person through skin contact.

Blistering agents

These were the first of the modern agents developed specifically for military purposes. Unlike nerve agents, blistering agents are different mixtures of one compound, sulfur mustard, with other non-toxic chemicals which affect its dispersion characteristics.

The primary hazard with sulfur mustard involves skin contact. The term “blistering agents” is somewhat of a misnomer - it will kill you if you inhale enough of it, but this is not likely to occur as it is not particularly volatile under normal conditions. Significant quantities of sulfur mustard (a.k.a. HD or HT) have been produced in the U.S.

Chemical Agents in the Environment

It is unlikely (but never impossible) that we at TestAmerica Sacramento will receive a sample that contains a dangerous concentration of active agent. We **should not** receive samples from any areas known to be contaminated with active chemical agent (areas with buried drums, old munitions, etc.) because the Army policy for such sites is to destroy the agent onsite.

Additionally, most of the areas in which degradate analysis is required are areas in which agent was used decades ago - all active agent is likely to have degraded. Nonetheless, it is imperative that we know as much as possible regarding the compounds and the samples in order to protect ourselves against any potential hazard.

The behavior of these compounds in the environment has been extensively studied. Most of the information we have is from a study titled **Environmental Chemistry and Fate of Chemical Warfare Agents**. This study was prepared for the Army Corps of Engineers by Southwest Research Institute in 1994.

Sites containing chemical warfare related material or chemical warfare materials are divided into “**stockpile**” and “**non-stockpile**” sites. Stockpile sites are where the vast majority of CWM’s are stored. Non-stockpile sites are where smaller amounts of CWM’s are located. Non-stockpile sites may contain: buried CWM, chemical weapon production facilities, binary chemical weapons and miscellaneous CWM.

What is important to realize is that based on the **Survey and Analysis Report** prepared by the US Army Chemical Material Destruction Agency (11/93), there are “potential burials at 82 locations in 33 states, the US Virgin Islands and the District of Columbia.....Some of the 82 locations have multiple burial sites.” Given this wide span of impacted areas, for every shipment received by TestAmerica Sacramento for CWM analysis, adherence to procedures listed in this document and the Environmental Health and Safety Manual is strictly required.

Nerve Agents

Both of the compounds we are concerned with (Sarin and VX) undergo **hydrolysis in the presence of water**. This hydrolysis proceeds at different rates, depending upon the compound. At worst (cool temperature, normal pH, no dissolved ions, no microbes), either of these compounds getting into water would be degraded to extremely low levels ($< 1 \times 10^{-6}$ of the original concentration) within a couple of years. It is more likely that this level of degradation would occur much more quickly.

Degradation in soil is a far more complex issue. The rate of hydrolysis will depend upon a variety of factors, including soil moisture, pH, mineral content, microbes, temperature, etc. Most available studies show that these compounds last no more than a few days in the tested soil types.

Sulfur Mustard

In some ways, sulfur mustard behaves in a fashion similar to the nerve agents - i.e. it hydrolyzes rapidly in the presence of water. There is, however, an important difference. Under the right conditions, **the hydrolysis products of mustard can polymerize and form bubbles containing active mustard**. The mustard inside of these bubbles is shielded from further hydrolysis by the hydrolysis products. This has turned out to be a problem in areas where large amounts of mustard were dumped at sea. Fishermen in such areas have been injured when pulling up nets contaminated with blobs of active mustard. An indication that this may have occurred would be a biphasic sample. This situation can also occur in soil.

Potential Hazards to TestAmerica Sacramento Personnel

Following steps listed in this appendix and other safety policies will help reduce hazards to the greatest degree possible. However, such policies are no substitute for educated, observant personnel.

As always, you must think about what you are doing when you handle these materials. No policy can account for every potential situation. Staff members are expected to follow all sample handling policies identified in the Corporate Safety Manual and steps following sample receipt listed in this appendix.

Soil and water samples will be screened for agent prior to their shipment to TestAmerica Sacramento, unless an exception has been granted by the Corporate Director of EH&S. Data should be reviewed at the project management and EH&S staff level to ensure samples are “safe” for handling. If the screening status is unknown (i.e. no data is available), project management personnel should be consulted. Samples will **not** be handled. If staff are unavailable, the cooler will be left in cold storage until the situation is resolved. The expiration of analytical holding times will not be considered as sufficient reason to handle/process CWM samples prior to receipt of screening data.

Positive “hits” on samples containing by-products of agents (like EA 2192) must also be reviewed at the project management and EH&S staff level to ensure samples are “safe” for staff to handle.

When in doubt, seek assistance from project management, operations manager, or EH&S staff. Corporate EH&S staff are also a resource which must be consulted before deciding to proceed with any “questionable” samples received.

Sample Receipt Procedures

- ⇒ Follow WS-QA-0003
- ⇒ Review screening data BEFORE opening the cooler if soil samples.
Note: Double gloves are required when handling chemical warfare degradate samples.
- ⇒ Following established safety policies, open the cooler, remove any paperwork, and check the interior condition.
- ⇒ All coolers from CWM sites will be initially opened in a fume hood. Once you have determined that there are no broken or leaking sample containers, the cooler may be moved to a bench top for further processing.
- ⇒ If a cooler contains a broken or leaking sample, isolate the cooler in the hood and IMMEDIATELY contact the project manager, operation manager and/or EH&S staff.
- ⇒ Any sample which appears to be biphasic in appearance must be isolated. Immediate notification to project management, operation manager and/or EH&S staff is required.
- ⇒ When in doubt, get help regarding sample receipt. Worker health and safety is **paramount** to sample analysis.
- ⇒ Regardless of screening data, any cooler containing samples for degradate analysis (or any other samples from an area suspected of potential agent contamination) **should be inspected carefully** upon receipt. Anyone inspecting the samples, logging them in, or handling them for any other reason should observe all of TestAmerica Sacramento's regular safety procedures.

In addition, **two pairs of gloves** will be worn in order to minimize any potential for skin contact with toxic compounds. Please note that **skin contact appears to be the most likely potential route of exposure**. This is based on the fact that nerve agents are likely to have degraded leaving sulfur mustard as the most likely potential contaminant and due to the likelihood that the samples will be cold. The temperature in the cooler is important from the standpoint of safety as well as sample integrity - cold samples mean a significantly lower potential for any kind of toxic vapor formation. Coolers containing broken jars or bottles should be placed in a hood immediately and left there until the client has been contacted. EH&S staff and/or the project managers will give instructions regarding return to client or disposal based on the screening data.

In conclusion, it must be emphasized that these samples must be handled with the appropriate level of care. Observant, educated personnel are our best defense against exposure to any kind of toxic materials found in our samples.

APPENDIX 8

Handling of Blood or Other Potentially Infectious Material

Background

TestAmerica Sacramento has, upon occasion, received a variety of biological samples for various environmental analyses. Biological samples present a very different type of hazard than “typical” environmental samples. Depending on the type of sample delivered for analysis, and the types of analyses requested, a variety of additional precautions and protective measures may be required when receiving, processing and storing these samples.

Types of Biohazard Samples

There are many types of biohazard samples. Not all biological samples are necessarily biohazard samples. Some of the types of biohazard samples that have been received at TestAmerica Sacramento in the past include:

- Human blood

- Human tissue

- Human breast milk

- Rodent or other mammalian tissue

- Human waste products, usually samples from municipal sewage treatment plants

While fish, crawfish, clams, plant tissue, grasses, and such are all biological samples, they are not generally considered to be a biohazard threat.

Specific Hazards Associated with Biohazard Samples

The unique threat associated with biohazard samples is infectious diseases. Typically, these are Human Immuno Virus (HIV) and Hepatitis B. Other potential hazards include (but are not limited to) rabies, bubonic plague and the Hanta virus. Some of these hazards we are prepared to work with effectively, and others we are not.

Samples potentially infected with bubonic plague or the Hanta virus requires engineering controls that are not in place at TestAmerica Sacramento. Accordingly, we will not accept samples potentially infected by these diseases. These samples include whole rodents or other mammals, mammal parts, or homogenized mammal tissue. Mammal tissue samples that are known NOT to be infected with these diseases may be accepted for analysis under certain conditions. These samples must be homogenized and the sample tissue must be “fixed” in a 4% or higher formalin solution. The outside of the sample container must have been disinfected with a Centers for Disease Control (CDC) approved disinfectant after the sample was placed in the container but before it was shipped to us. Examples of this disinfectant solution are a 10% bleach solution or a 5% Lysol solution.

General Procedures

Universal precautions: All human and mammal blood, fluid and tissue samples are assumed to be infectious. All staff members will wear two pairs of protective gloves when handling or working with biohazard samples. Safety glasses and a face shield are required. Fume hood sashes will be closed as far as possible, consistent with safe work practices. Lab coats will always be worn and buttoned up. Lab coats worn when handling biohazard samples will not be worn outside of the laboratory. When work is finished with biohazard samples, lab coats worn during the process will be sent out for cleaning. If they have been splashed or contaminated with any infectious sample, they will be disposed of as biohazardous waste. Workers will exercise caution to avoid injury with tools possibly infected from biohazard work, such as glass pipettes, metal spatulas, broken glass, etc. All waste material will be disposed of in appropriately marked containers as biohazardous waste. Workers with open wounds, sores or broken skin shall not handle biohazardous samples. Pregnant workers shall be especially familiar with and adhere to precautions to minimize the risk of transmission. Employees involved with handling human blood or tissue samples will be offered the opportunity to receive the Hepatitis

B vaccination series. This may be accepted, declined, or accepted at a later date.

Engineering and administrative controls: Signs will be posted on all doorways leading into areas where biohazardous samples are being handled. These signs will be clearly visible and will identify that biohazard work is in progress. When these signs are posted, personnel not involved in the work will stay out of the work area. If this is impractical, the biohazard work will be performed in an isolated area that is clearly marked. No one may enter this area without permission from the sample administration technician or chemist doing the work. Personal protective equipment will be removed immediately upon leaving the area and disposed of or cleaned properly. Eating, drinking, use of tobacco products, gum, hard candy, applying cosmetics or lip balm and use of contact lenses are all prohibited in any areas where biohazard samples are being handled. Employees working with biohazard samples will thoroughly wash their hands with disinfectant soap when finished work and before leaving the lab. Work areas will be thoroughly cleaned and disinfected when biohazard work is complete. This includes properly disposing of bench paper and used equipment such as pipets, disinfecting all reusable equipment such as glassware, metal spatulas, and disinfecting work surfaces. Broken glassware that is potentially infected must not be picked up directly with the hands. Any trash cans or containers that may have been contaminated will be inspected, cleaned, and disinfected with an appropriate disinfecting solution.

Sample Receipt Procedures

Follow WS-QA-0003

If advance notification is provided of incoming biohazard samples, contact EH&S, review this appendix and ensure that you are familiar with the safety procedures involved. Ensure that you have a clear workspace, that you know in advance where the samples will be stored, and that there is space available to store them.

Note: All potential biohazard samples must be kept in locked storage - either WR1 or WF1.

Note: Double gloves and a face shield are required when handling biohazard samples.

Biohazard samples shall be opened in a fume hood.

Following established procedures, open the cooler, remove any paperwork and check the interior condition.

If a shipping container has a broken or leaking sample, isolate the cooler in a fume hood and IMMEDIATELY contact the project manager and EH&S staff.

Note any comments or warnings on sample containers (including shipment paperwork) regarding specific threats or hazards.

When in doubt, get help regarding sample receipt. Your health and safety is of paramount importance.

The most likely methods of transmission of disease when handling biohazard samples are splashing infected blood or tissue onto an open cut or sore or into your eyes, mucous membranes or mouth. The likelihood of transmission via these routes can be almost completely eliminated by following proper procedures.

Exercise care when handling samples so that they do not drop or get knocked over.

Wear two pair of protective gloves – latex, vinyl or nitrile.

Don't work around biohazard samples with open cuts or sores.

Wear your safety glasses with a face shield.

Ensure that all skin is covered, such as your wrists and forearms

Wear your lab coat, properly fastened.

APPENDIX 9

PFAS Bottle Prep and Sample Handling

- Wash hands and put on proper PPE (gloves).
- Avoid plastic clipboards, binders, glues, and adhesives.
- Never eat or drink while preparing bottle orders.
- Avoid all Teflon-containing materials while preparing bottle orders for PFAS.
- Line carts with clean bench paper or paper towels before placing PFAS containers in them.

If your PPE was used in conjunction with glass containers, low density polyethylene (LDPE) materials, Teflon-lined caps, or blue ice packs, wash your hands and then replace it with clean PPE. Make sure your work area (bottle prep cart, work bench) has been cleaned well and that bench paper has been replaced before starting work again.

If there are contamination issues, the following materials used for apparel and footwear are not recommended to be worn when preparing bottles:

- New cotton clothing
- Any water resistant, waterproof, or stain-treated clothing,
- Any clothing containing Gore-Tex™
- Any clothing laundered using fabric softener
- Boots containing Gore-Tex™
- Tyvek®

Additional Precautions: PFAS-free deionized water for field blanks is taken from the water polishing systems in GenChem or Metals. Be sure to not let the tubing touch the water in the containers when filling the containers.

APPENDIX 10

Sample Receipt and Procedures Training Guide

NOTE: These are general guidelines. See specific client instructions for projects that are handled differently than the below procedures.

1. Remove and save custody seal(s), if present. Record on the Field Sheet (if a TestAmerica custody seal, record the seal number).
2. Remove and save Waybill, if present, and record the number on the Field Sheet.
3. Find Temperature Blank, if present.
4. Take and record cooler temperature.
5. Sign and date the Field Sheet.
6. Sign, date, and record receipt time, cooler temperature(s), coolant type, and thermometer used on the COC.
7. Remove samples from the cooler. **IMPORTANT: If more than one cooler per COC is used, especially if there is a temperature excursion, find or create a cooler ID and mark the cooler ID on the sample containers from that specific cooler.**
8. If the container types are not obvious on the COC, fill out a Bottle Lot Inventory Sheet.
9. If the project is designated a DOD or DOE project and preserved containers are present, check the pH of the containers and record the pH on a Sample Receiving Preservation Check sheet.
10. If VOA vials are present and not in Ziploc bags, place them in Ziploc bags.
11. Place samples into the walk-in refrigerator WR-2. Exceptions: if frozen, place into freezer F-10; if a sample is product and/or if the sample has an obvious odor, and the samples needs to be kept cold, place into refrigerator R-22. Write the exception location on the Field Sheet.
12. Make a photocopy of the Custody Seal(s) and Waybill; if other cooler labeling is present, photocopy this as well.
13. Place signed COC, signed Field Sheet, photocopies, original cooler labeling, and any other paperwork accompanying the samples into a manila folder for standard TAT requests or a red folder for rush TAT requests. If subcontracted analyses are requested and identified place the manila or red folder into a blue folder. Place the folder in the rack for login.
14. If soil samples were in the cooler, dump the ice into the Sample Receiving sink. Place a red "Soil Sample" sticker onto the lid of the cooler.
15. If only water samples were in the cooler, the ice may be dumped down an outside storm drain if room in the Sample Receiving sink is limited.
16. Remove and throw away all remaining client stickers and tape from the cooler. Place the cooler into one of the cooler cages for processing.

APPENDIX 11

TALS Checklist Training Guide

Question	Answer	Notes when trained
Radioactivity wasn't checked	NA	
Cooler Seal	Yes	This is all 3: Yes a seal with no info, add seal # or NA (no seal)
Sample Custody Seal	NA	Most likely going to be NA, some samples from Alaska will be sealed
Cooler not tampered with	Yes	Note/NCM if tape or seal is torn or missing
Sample received on ice	Yes	Answer: Yes or NA (if Air)
Cooler temp acceptable	Yes	Yes or NA (if Air)
Cooler temp recorded	Yes	Answer: Yes or NA (if Air). Some air will be temped.
COC Present	Yes	This is Yes or No
COC is filled out in ink	Yes	
COC is filled out with all information	Yes	Failures notes will say "COC was not relinquished."
Field sampler present	Yes	Answer: Yes or No -> for SUB ICOCs, in failure reason: Received project as a subcontract.
No discrepancies between containers & COC	Yes	Answer Yes or No. If no, write a brief note about what was wrong or enter the failure reason
Within Holding time	Yes	Answer: Yes or No
Legible labels	Yes	Answer is very rarely No.
Not broken or leaking	Yes	Answer: Yes or No. Check the failure reason.
Date/time provided	Yes	Answer is No if the COC is missing date OR time. Check samples for info as well.
Appropriate containers	Yes	Answer is very rarely no.
Sample bottle completely filled	Yes	Answer: Yes or No. If perchlorate, use this statement: No, headspace for 314/331/6850 (headspace required for these methods).If air, NA .
Sample preservation	NA	
Enough volume for MS/MSD	Yes	If Air, NA .
Zero headspace / no headspace or bubbles	Yes	This is used only for VOA vials. If Air, NA .
Multiphasic samples not present	Yes	
No splitting or compositing	Yes	
Residual chlorine checked	NA	

APPENDIX 12**After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel**

Normal business hours for receiving samples are Monday through Friday, 8:00 am to 6:00 pm, and Saturday, 8:00 am to 12:00 pm. In the event that samples are delivered outside of normal operating hours, and only non-Sample Administrative personnel are available to accept the delivery, the following procedures should be followed:

- If samples are delivered directly by a client, have him/her relinquish the COC, sign your name on the “Received By” line, note the time received, and make a copy of the signed COC for the client. Keep the original COC with the samples.
- If samples are delivered by a courier, and the COC is taped inside a cooler, note who delivered the samples and what time they arrived on a “Notes Form” (QA-812, an example is in Appendix 11 of this SOP).
- Record the date/time received and the custody seal IDs on the “Notes Form”. Open the cooler/container and measure the temperature of the samples and/or temperature blank (if easily accessible) using the IR or AK thermometer. Do not take the temperature of ice or packing material. Record the temperature(s) on the form.
- If samples are not contained in a cooler, document what kind of cooling agents were used, if any, on the Notes Form. Put the form with the COC.
- Place cooler/samples on a cart and store in the walk-in refrigerator.
- Send an email to “SACSC” to notify them that samples were received outside of normal hours, and include any pertinent information (i.e. when received, who delivered the samples, where they are located, sample receipt temperatures, etc.) to assist them in processing the samples when they return.

Facility Distribution No. _____

Distributed To: _____

APPENDIX 14
Canister Receipt Notes

CANISTER RECEIVING

Canister ID	Flow ID	Canister ID	Flow ID
1	1	16	16
2	2	17	17
3	3	18	18
4	4	19	19
5	5	20	20
6	6	21	21
7	7	22	22
8	8	23	23
9	9	24	24
10	10	25	25
11	11	26	26
12	12	27	27
13	13	28	28
14	14	29	29
15	15	30	30

325B Pelican Case Inventory

Case ID: _____ # of Filter Caps: _____ Zip Seal: _____
 Temperature: _____ # of Sample Tubes: _____
 # of Wrenches: _____ # of Unused Tubes: _____
 # of Gloves: _____

Sacramento



Sample Receiving Notes - Job

Service: FedEx UPS Lab Courier Client Drop Off P.O. Std. Overnight 2-Day Ground Other: _____ Tracking #: _____ _____ _____ Notes: _____ _____ _____	Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC. Cooler Custody Seal: _____ Transferred by Sacramento - Yes <input type="checkbox"/> No <input type="checkbox"/> # Bags: _____ 1L, _____ 2L, _____ 10L # Canisters: _____ 1L, _____ 6L, TA <input type="checkbox"/> Non TA <input type="checkbox"/> # Canisters Unused: _____ 1L, _____ 6L # Co-Locators _____, # Gauges: _____ # Flow Regulators: _____ Initial & Date _____
--	---

APPENDIX 15

Revenue Source Tracking



Corporate Quality
Work Instruction for
Laboratory / Service Center Operations

This Work Instruction is to be attached as an Appendix to Location-Specific Sample Receiving and / or Shipping Standard Operating Procedures (SOPs).

> This Work Instruction is in effect as of 10/1/2018 in all Locations. <

The purpose of this Work Instruction is to provide instructions to be followed in the gathering of data to track revenue sources from service centers and transitioning from re-missioned locations. This information is collected from the processes employed in sample shipment and receiving.

Stamp Generation

Stamps should be used at all service center locations, including those labs that house service center functions.

Some locations will have multiple stamps to allow for multiple aspects of tracking. For instance, Boston will have two label types: Boston and Boston / Westfield.

When a location needs a new self-inking stamp, they should enter a requisition in Oracle to Office Depot for item 666632 and customize with the service center name and number.

Service Center Responsibility

Each service center will be responsible for affixing a stamp on the COC.

Labels will no longer be placed on the coolers.

Where two locations are involved in providing support, the following is the order of priority for which service center stamp should be on the COC:

1. Location that picked up the samples from the client (provided logistical support)
2. Location that dropped off the bottle kit / cooler to the client (provided logistical support)
3. Location that shipped bottles to the client (in lieu of a lab shipping bottles)

While it isn't common for one service center to provide bottle kits, and a second service center to pick up samples, it can happen. The service center picking up the samples should single line cross out and initial the initial service center label while affixing their stamp to the COC.



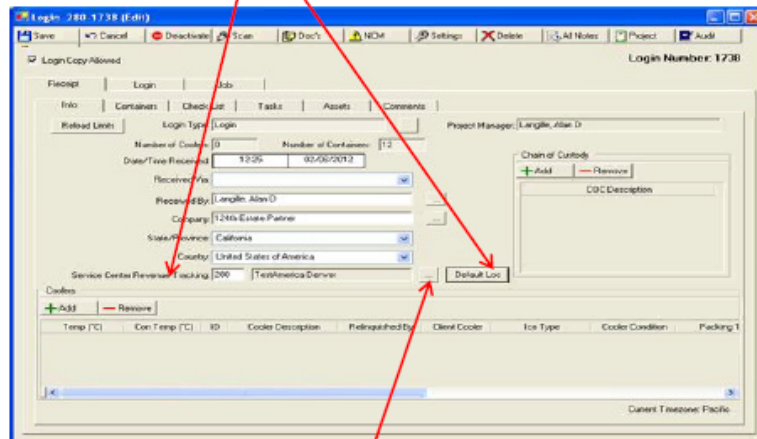
Corporate Quality

Work Instruction for
Laboratory / Service Center Operations

Lab / Sample Receiving Responsibility

For every log in, sample receiving personnel are responsible to use the drop down menu and enter the stamp from the COC, which will enter the "Service Center Revenue Tracking" into TALs.

If there is no stamp on the COC, sample receiving will select their home location by using the "Default Loc" button.



The service center location must be selected using the list feature in TALs if it does not automatically fill in when the project is loaded.

As of July 1, 2018 this data will be considered mandatory in TALs. The login cannot be saved successfully until the Service Center Tracking field information has been completed.



Corporate Quality

Work Instruction for
Laboratory / Service Center Operations

Project Manager (PM) Responsibility

The PM's are required to identify the "source" of revenue on the project, or job which will be located on the COC. For example, if a project was previously handled in Westfield but has transitioned to Buffalo, the PM should select the appropriate *Service Location* as Westfield on the Project screen.

The screenshot shows a software window titled 'Project - 36932245'. The interface includes a menu bar (New, Edit, Copy, Delete, Paste, Print, Find, Docs, Notes, Project, Hot Links, Audit) and a toolbar. Below the toolbar, the project details are displayed:

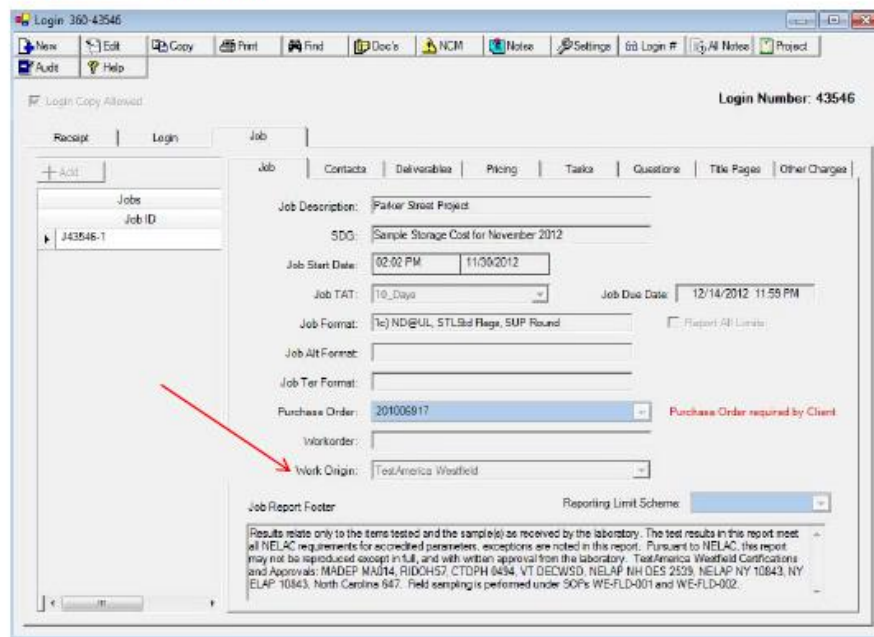
- Number code: 36932245
- Location: 360 TestAmerica Westfield
- Company: JANEK Environment & Infrastructure, Inc.
- Details: Contacts | TATs | Tasks | Deliverables | Other Charges | Analysis Groups | Analytical Units | Action Links | Action List Groups | Sites/Events | Bottle Orders | Quote Info
- Menu: Main | Quote
- Location: 360 TestAmerica Westfield
- Number Code: 36932245
- Description: Foster Street Project
- Market Segment: Commercial - Unknown
- Service Location: TestAmerica Westfield
- Default PO: 201306917
- Default WO: [Empty]
- Project Manager: Hobart, Paul
- Project Mgr Assistant: [Empty]
- Account Executive: [Empty]
- Project Start: 08/30/2010
- Sample Check List: Standard Sample Receipt Checklist
- TAT Calculation Type: Business Days
- Turn Around: 10_Days
- Method of Disposal: Disposed by Laboratory
- Contract ID: [Empty]
- Billing Cust Code: [Empty]
- Payment Terms: [Empty]
- Minimum Invoice Amt: [Empty]
- Expected Quote Value: [Empty]
- Gov't Designation: N/A
- Status: Active
- Condition: Active
- Original Copy: [Empty]
- Program: [Empty]
- Project: [Empty]
- Quote: [Empty]
- Report Footer: (Results relate only to the items tested and the sample(s) as received by the laboratory. The test results in this report meet all NELAP requirements for accredited parameters. Exceptions are noted in this report. Pursuant to NELAP, this report may not be reproduced except in full, and with written approval from the laboratory. TestAmerica Westfield Certifications and Approvals: MADEP MA214, RIDGHS7, CTDPH 0494, VT
- TAT Price Mult: 1.00000
- Disposal Days: 45
- Reporting Limit Scheme: [Empty]
- Formatter: (c) ND@UL STLBD Rspg, SUP R; [Empty] Alt [Empty]
- Total Reference Amount: \$2,723.00
- Total Amount: \$2,723.00
- Total Discount Pct: 0
- Total Plus Allow Ref Price: 0



Corporate Quality

Work Instruction for
Laboratory / Service Center Operations

The PM's may become aware of the "source" of revenue sometime after the project build is complete. The information can also be updated on the Job tab of a login, but the same data field is referred to as Work Origin on the Job tab of a login:



The screenshot shows a web browser window with the TestAmerica interface. The 'Login' tab is active, and the 'Job' sub-tab is selected. The 'Work Origin' field is highlighted with a red arrow. The 'Purchase Order' field is also visible and set to '201006917'. The 'Job Description' is 'Falkner Street Project' and the 'SDO' is 'Sample Storage Cost for November 2012'. The 'Job Start Date' is '02/02 PM' and '11/09/2012'. The 'Job TAT' is '10_Days' and the 'Job Due Date' is '12/14/2012 11:59 PM'. The 'Job Format' is '7c) ND@UL, STL@id Flaga, SUP Round'. The 'Job Alt Format' and 'Job Test Format' are empty. The 'Reporting Limit Scheme' is set to a dropdown menu. The 'Job Report Footer' contains a disclaimer about NELAC requirements and field sampling procedures.

Invoices associated with projects / jobs / logins that are tagged with this information will be tracked as being sourced from a service center and / or re-mission location. It is the responsibility of PM, Service Center and Sample Login staff to ensure every effort is made to properly identify the "Source Location" for all revenues.

APPENDIX 16 Sample Receipt and Procedures Checklist

Sample Receipt and Procedures Checklist

Note: this is a general guideline checklist. See specific client instructions for projects that are handled differently from the below procedures.

- Remove and save Custody Seal(s), if present; record on the Field Sheet (if a TestAmerica Custody Seal, record the seal number).
- Remove and save Waybill, if present; record the number on the Field Sheet.
- Find Temperature Blank, if present.
- Take and record cooler temperature.
- Sign and date Field Sheet.
- Sign, date and record receipt time, cooler temperature(s), coolant type and thermometer used on COC.
- Remove samples from the cooler. **IMPORTANT: if more than one cooler per COC is used, especially if there is a temperature excursion, find or create a cooler ID and mark the cooler ID on the sample containers from that specific cooler.**
- If the container types are not obvious on the COC, fill out a Bottle Lot Inventory sheet.
- If the project is designated a DoD or DOE project and preserved containers are present, check the pH of the containers and record the pH on a Sample Receiving Preservation Check sheet.
- If VOA vials are present and not in Ziploc bags, place them in Ziploc bags.
- Place samples into the walk in refrigerator WR-2. Exceptions: If frozen, place into freezer F-10 (any sample with potential of breakage is to be placed into a Ziploc bag for secondary containment). If a sample is product and/or if the sample has an obvious odor, and the sample needs to be kept cold, place into refrigerator R-22. Write the exception location on the Field Sheet.
- Make a photocopy of the Custody Seal(s) and Waybill. Photocopy any other cooler labeling present.
- Place signed COC, signed Field Sheet, photocopies, original cooler labeling and any other paperwork accompanying the samples into a: manila folder for standard TAT requests, red folder for rush TAT requests. If subcontracted analyses are requested and identified, place the manila or red folder into a blue folder. Place the folder in the rack for login.
- If soil samples were in the cooler, dump the ice into the Sample Receiving sink. Place a red "Soil Sample" sticker onto the lid of the cooler.
- If only water samples were in the cooler, the ice can be dumped down an outside storm drain if room in the Sample Receiving sink is limited.
- Remove all remaining client stickers and tape from the cooler.
- Place the cooler into one of the cooler cages for processing.

APPENDIX 17
Terra Cores InstructionEnvironment Testing
TestAmericaSacramento
Terra Cores Instruction**Terra Cores**



In the Terra Core kit there will be a foam holder with
3x 40mL VOA Vials, 1x 20ml polyethylene bottle and 1x 4 oz or 8 oz glass jar.
(note: the 3 vials may also come in a zip lock bag, **DO NOT** take them out of this bag.)

The 3 vials are 2x 40mL VOA Vials w/5mL NaHSO₄ and 1x 40mL VOA Vial
w/5mL MeOH.

**When we get Terra Core kits DO NOT label the
3x 40mL VOA Vials in the kit!**

OK to label the 4oz or 8oz jars and 20mL bottle.

If you have any questions on how to proceed, contact a member of the Sample
Receiving staff.

	Always check on-line for validity. <h2 style="text-align: center; color: red;">Automated Storage, Retrieval, and Discarding of Samples</h2>	Level:  Work Instruction
Document number: S-SS-WI12042		
Old Reference: 1-P-QM-PRO-9015512; DOD - SOP-SS-006		
Version: 11		Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date 28-JUN-2019	Document users: 5_EUUSLA_Sample Support_Manager, 6_EUUSLA_Sample Support_ASRS	Responsible: 5_EUUSLA_Sample Support_Manager

Note: Handling of Quarantine Soils approval is through Quarantine Soil Permit P525-190620-001 - Robert Dempsey (ELLE President), permittee

[Revision log](#)
[Reference](#)
[Purpose](#)
[Scope](#)
[Safety Precautions and Waste Handling](#)
[Personnel Training and Qualifications](#)
[Procedure](#)

Revision log

Revision: 11		Effective Date: <u>This version</u>	
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure B	Applicable to the procedure	Added references to the scanned locations	
Procedure G	Changes made to the process	Updated the hazardous waste stream information appearing on screen Updated the chart. Changed the Acid Waste, Hazardous & Quarantine Soil Wastes, and Soil Extracts in Sodium Bisulfate descriptions	

Revision: 10		Effective Date: <u>Mar 15, 2017</u>	
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure B.3.	Clarification	Added location of samples with Cart ID	
Procedure B.7.	Enhancement	Added the Missing Samples-Requisition Contacts email information	

Reference

Chemical Hygiene Plan, Lancaster Laboratories, current version.

Purpose

The purpose of this SOP is to define correct procedures for the storage, retrieval and discard of samples using the Automated Storage and Retrieval System (ASRS) or the manual Sample Storage program.

Scope

This SOP covers the steps required to put samples in location, pull samples for analysis, and locate samples not found in the ASRS. It also outlines the procedures used for handling samples designated as “Hold at Discard” and removing samples upon their discard date for disposal.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal and state laws and regulations.

Information concerning the known toxicity, properties, or any special handling precautions can be found in the material safety data sheets (MSDS) available in Sample Storage or by a Safety Officer. The MSDS should accompany the samples when pulled for the technical departments. Safety glasses and sample storage lab coats are required as personal protective wear.

Hearing protection devices (earplugs) are available. However, occupational noise measurements indicate that the sound pressure level is below the Occupational Safety and Health Administration’s (OSHA) Action Level of 85 dB (A). The Action Level is the threshold above which OSHA mandates hearing protection.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

The initial training consists of observing all of the steps in this procedure carried out by an experienced ASRS operator. Next, the trainee performs all of the same steps while the experienced person watches, answers questions, and gives feedback. Following the initial training, an experienced operator is available as a resource until no longer required. Operators are considered proficient when all of the steps in the procedure can be carried out independently.

Procedure

A. Daily ASRS operations/pulling requisitions:

1. Log onto the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Choose the *Pick* option in the ASRS Functions.
4. Requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled.

Choose the *Shipping* option on the Function Selection Panel and then select *Order Wave Release*, the rule that best describes the orders you wish to pull, and change the Order Status to *Batched*.

5. To release orders, select the *GO* option on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. Place the Tote ID with the order so the samples can be scanned to this ID.
6. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples to fill the order are displayed in green; scan the Sample Bar Code and the Tote ID to remove the sample from the container and link it to the designated Tote ID.

NOTE: Discards can also be done at this time. See Procedure Section E.

7. When an order is finished, missing samples are displayed on a Discrepancy List found in Parallax. Any other queued orders are also processed.

8. Completed orders are stored in the appropriate queuing area until a lab technician comes to retrieve them.

B. Locating samples not in the ASRS

1. When all of the samples are retrieved from the ASRS, check the Discrepancy List to view any samples that are still needed to complete the order.

2. Samples previously assigned to another Tote ID are located on a cart waiting to be put back into the ASRS, or have not yet been returned from the lab.
 3. Samples with a Cart ID, Bin ID, or room ID are scanned to a fixed location and can be located in the designated soil location area, in the associated laboratory fixed location, or at one of the splitting stations. Overflow carts can be found in the ASRS walk-in.
 4. If no prior destination is given, the samples are new and should be located in the SA walk-in waiting to be labeled or processed by one of the other sample support areas. If the samples were already processed by one of the other areas, the samples should be on a ASRScart waiting to be scanned to a location cart and put back into the ASRS.
- NOTE:** Samples that require a pH check, volatile prep, or homogenization must be communicated to that area prior to the samples being given to the technical areas.
5. Samples that are not located by the requisition time need to be communicated to the appropriate lab technician. Keep the technical department up-to-date on the status of missing samples. If the technician informs you that a sample is *Rush* status, the sample becomes a priority. If you require further assistance to find the sample, contact your supervisor.
 6. If the sample is not located by the end of a shift, the ASRS operator is required to let the next shift know the status of the sample. If the sample is missing on your next shift, you must contact your supervisor.
 7. If a sample is not found 24 hours prior to the deadline, the client service representative must be contacted to inform the client. These samples are indicated by the labs using the Missing Samples-Requisition Contacts email address which alerts this group that the sample must be located or a reason must be provided as to why the sample was not found.

C. Retrieving samples designated as client hold

1. Processing the Client Hold Orders
 - a. Log on to the system by typing your employee login and password.
 - b. Choose the *ASRS* option in the Function Panel
 - c. Choose the *Pick* option in the ASRS Functions
 - d. Client Hold requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled. This is done by choosing the *Shipping* option on the Function Selection Panel. Then select *Order Wave Release*, chose the *Client Hold* orders, and change the status of the orders you wish to pull to *Released*.
- NOTE:** For Client Hold orders the *CLH* option must be selected as the conveyance type.
- e. When orders are released, the *GO* option is selected on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. The Tote ID is placed with the order and the samples are scanned to this

ID. If multiple orders for the same client are placed in the same tote all Tote IDs must be displayed.

f. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples needed to fill the order are displayed in green. Scanning the Sample Bar Code and the Tote ID removes the sample from the container and links it to the designated Tote ID. If a container is filled before the order is complete you can request a new Tote ID by scanning the next sample, using the mouse and right clicking on the proper order on the Put Bar and select *New Tote ID*.

NOTE: Discards can also be done at this time. See Section E.

g. When the order is finished, the box containing the samples must be taken over to the bulk workstation. There the samples are imported into the Client Hold area by inducting the Tote into an available location.

2. Scanning Totes in to the Client Hold Pod

a. Using the RF gun log in to the system.

b. On the Main Menu choose the *Ship* option.

c. Next choose *Matrix Induct*.

d. Scan the Tote you wish to put away and scan an Empty shelf location in the Client Hold Pod. You will get a question to Consolidate “Yes or No”. Always answer “Yes”.

e. If multiple Tote IDs are on the current box, scan each ID to the same location. The last ID scanned contains all the samples in the box. All previous IDs scanned must be crossed off with a sharpie so that only the last ID scanned is displayed.

NOTE: If a container is scanned to a location that is not full and has the same client and account number as a Tote you are currently scanning away, you can combine by:

f. Repeating steps 1 – 3 above.

g. Scan the Tote you wish to combine to the location in which the container resides.

h. All the samples from the existing container are transferred to the container ID that was recently scanned to the location; all samples need to be moved to the current box.

3. Discarding from containers in the Client Hold Pod

a. Remove the container from the designated location and write the shelf location on the container ID (this is to allow for the container to be returned to the proper location if all the samples aren't up for discard at this time).

b. Using the Evolution workstation, choose the *Bulk* option on the Function Selection Panel.

- c. Navigate to Execute and Deplenish Container.
- d. Choose the Existing Container option.
- e. Scan the container ID.
- f. Scan each sample located in the container. There are three scenarios that could take place:
 - (1) Sample is obsolete - remove from the container as normal and discard.
 - (2) Sample is obsolete and hazardous - remove from container and place on a designated hazardous cart.
 - (3) Sample has not reached the necessary time to be marked as obsolete and remains a client hold. In this case the system prompts you to scan a container. The options are:
 - (a) Scan the existing container. All samples designated as client hold remain in the current container.
 - (b) Scan an existing container that is scanned to a different Client Hold Location. All samples designated as Client Hold move to this container (samples must be same client and account number).
 - (c) With either option the container scanned remains unless a new container to discard from is selected.
- g. Return the container to the designated shelf location if samples remain. If no samples remain the container ID must be removed and the box is not returned to the Client Hold Pod.

D. Sample put away

1. Log on to the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Select the *Put Away* option in the ASRS Functions.
4. Choose *Put Away*, which displays the available empty locations for each cell configuration listed in order of container height.
5. Choose the container size you need and type the number of samples and press ENTER. This brings you the available containers for the size requested.
6. When the containers arrive, scan the container ID. The grid display on screen shows the available open locations.
7. Scan the Sample ID and the Open Location ID to code the sample away.

NOTE: Procedure D. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

E. Sample discard procedure

NOTE: Discards can be processed through Parallax. In the SA function, choose *Download ASRS Discard File*. This starts a count of the samples that are up for discard and sends the file to *Evolution*.

1. Log on to the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Select the *Deplenish* option in the ASRS Functions.
4. Select the *Discard* option which displays the available discards for each cell configuration listed in order of container height.
5. Choose the container size you wish to discard from and type the number of samples you need and press ENTER. This brings you the available containers for the size requested.
6. When the containers arrive, scan the container ID and the grid displays on the screen. The regular obsolete samples are displayed with blue lines going through the sample; hazardous are in red.
7. Scan the designated samples and separate upon removal from the ASRS and take to the appropriate area for disposal.

NOTE: Procedure E. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

F. Disposing non-hazardous samples

1. Use discretion when disposing all non-hazardous samples. Do NOT discard a solid or liquid sample if it has an “off odor” or unusual appearance.
2. Liquid samples should essentially be clear and odorless like water. It is acceptable for the sample to have some discoloration and/or sediment present. Unless the sample has a solvent odor, an oily or viscous appearance, a paint odor or appearance, a lot of solids, etc., it may be poured into the discard sink. The ventilation hood and the cold water tap must be turned on. After pouring the sample out, place the container in the trash hopper.
3. Discard solid, non-hazardous samples into the trash hopper. Do not dump solid samples out of their containers. Discard containers unopened, with the contents intact, directly into the hopper.
4. If you have any doubt on the proper disposal of a sample, place it on the cart designated for questionable discard and notify your supervisor or the EHS (Environmental Health and Safety) Group for assistance.

G. Disposing of hazardous samples

1. Take the hazardous discard cart to the Sample Storage workstation.

2. Log on to Parallax.
3. Using the SA function on the tool bar, choose the *Sample Storage* option.
4. When in this program, choose *Hazardous Discard Assessment*.
5. Scan the barcode on one of the hazardous samples.
6. If the sample discard date has not come up yet, a window appears indicating "Sample has not been discarded." If applicable, the hazardous results are displayed. Before disposing, further investigation must be done to see if the sample can be discarded.
7. If the sample does **not** have any Final Results that exceed the adjusted Hazardous Limit for that particular analysis, a window appears indicating "**No hazardous analysis in this sample.**" Read the lab notes very carefully at this point because the sample may be hazardous for reasons other than the analyses performed.
8. If the sample has no hazardous analyses or lab note indicating hazard, check the matrix of the sample and the sample designation to be sure that the sample matrix is not something that must not be dumped down the sink (i.e., oils, paint, or paint thinner, ink, etc.).
9. If the sample is flagged as *Hazardous* and the discard date has passed, the following information appears on the screen:

Master Analysis Number
 Piece Analysis Number
 Analysis Name
 Final Result
 Reporting Units
 Discard Date
 Adjusted Hazardous Limit

Waste Stream

10. Based on the data provided from the Assessment Program, the sample needs to be put into one of the appropriate waste streams which are indicated in the program and have fixed locations assigned in the hazardous assement area.

Waste Stream	Description
Acid Wastes	pH <4; no heavy metals; dumped in acid neutralizer

Waste Stream	Description
Solvent Wastes	A liquid flagged as hazardous due to an analysis result regulated under TCLP Waste Characteristic List (except heavy metals); dumped in solvent waste drum
Labpack Wastes	A solid or liquid flagged as hazardous that does not fit into one of the other categories
Hazardous & Quarantine Soil Wastes	A soil from USDA regulated counties(soil)
Waters containing Heavy Metals (free liquid)	Liquid flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver); liquid poured off into a drum
Waters containing Heavy Metals (in vials)	Liquid samples in vials flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver)
Waters containing Cyanides	Sample vials flagged for cyanide-containing compounds
PCB-containing Soils	Solid samples (and containers) flagged for PCBs
PCB-containing Water & Oils	Liquid samples (and containers) flagged for PCBs
Soil Extracts in Methanol	Place vials in open-head drum labeled: "GC/HPLC and 40-mL vials containing organic solvents"
Soil Extracts in Sodium Bisulfate	Place vials in open-head drum labeled: "Corrosive Organic Extracts"



11. If you have any doubt on the proper waste stream for disposal of a sample, place it on the cart designated for questionable hazardous discard and notify your supervisor or the EHS (Environmental Health and Safety) Group for assistance.

12. Lancaster Laboratories reserves the right to return samples to the submitting client if they contain high levels of hazardous substances or do not fit into one of our existing waste streams.

End of document

Version history

Version	Approval	Revision information
9	22.APR.2011	
10	01.MAR.2017	
11	28.JUN.2019	

	Always check on-line for validity. <p style="text-align: center;">Legal Chain-of-Custody Documentation</p>	Level:  Standard Operating Procedure
Document number: QA-SOP11914		
Old Reference: 1-P-QM-QMA-9017335; LOM-SOP-ES-234		
Version: 3		Organisation level: 5-Sub-BU
Approved by: UDM6 Effective Date 15-MAR-2019	Document users: 4_EUUSLA_ELLE_All_Support, 4_EUUSLA_ELLE_All_Technical	Responsible: 5_EUUSLA_Env Quality Assurance_All

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[Purpose](#)
[Scope](#)
[Definitions](#)
[Personnel Training and Qualifications](#)
[Procedure](#)
 A. External COC documentation
 B. Internal COC Documentation Requirements
 C. Master List and Original Sample COCs
 D. Sample Custody in Technical Groups
 E. Completion of the process

Revision Log

	Revision: 3	Effective Date: This version
Section	Justification	Changes
Revision Log	Formatting requirement per G-DC-SOP16244	Removed revision logs up to the previous version
Throughout	Enhancement	Updated to D4 numbers and added form titles. Updated Figure references.
Figures	Fig 2 was already in Cross References	Removed old Fig 2. Figures 3 and 4 which contained examples were updated to Fig 2 and 3.
Procedure	Enhancement	Re-wrote Procedure for readability

	Revision: 2	Effective Date: Jul 6, 2015
Section	Justification	Changes
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version

Throughout Document	Reflects re-identification of documents in EtQ Company name change	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers Update all references to Lancaster Laboratories Inc. or LLI to be Eurofins Lancaster Laboratories Environmental or ELLE
Cross Reference and Procedure NOTE	Reference to overall system SOP not needed	Removed references to LOM-SOP-LAB-220 as this is an overall lab procedure that must be used in all documentation. A specific reference to it is not necessary.
Figures 1 and 2	Form were outdated	Replace with latest version of the form
Figures 3 and 4	Reflects current process	Replace with current example

Reference

Environmental Quality Policy Manual, Eurofins Lancaster Laboratories Environmental (ELLE), current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6874	Secure Storage Chain of Custody Original Sample
S-SA-FRM10722	Environmental Sample Administration Receipt Documentation Log
Q-EQA-FRM6862	Department Storage Chain of Custody Metals
Q-EQA-FRM6854	Secure Storage Chain of Custody Leachates
Q-EQA-FRM6864	Department Storage Chain of Custody
S-SS-FRM10672	Chain-of-Custody Transfer Record
Q-EQA-FRM6865	Secure Storage Chain of Custody Supplemental Information
Q-EQA-FRM6868	Secure Storage Chain of Custody Subsample
S-SA-FRM10731	Master List of Chains of Custody
QA-SOP11182	Sample Requisition
QA-SOP11184	Laboratory Sample Analysis Record (LSAR) Documentation

Purpose

In order to demonstrate reliability of data which may be used as evidence in a legal case, an accurate written record tracing the possession of samples must be maintained from the time they are received at the laboratory until the last requested analysis is verified. The purpose of a Legal Chain of Custody (COC) is to ensure traceability of samples while they are in the possession of the laboratory.

Scope

This procedure applies to the Eurofins Lancaster Laboratories Environmental (ELLE) and describes the Legal Chain of Custody (COC) documentation. The Legal COC is only initiated upon special request from the client. It may be requested if the samples are related to litigation. The Legal COC requires a hand-signed hardcopy COC document to be completed with every step from sample entry, sample storage, requisition, preparation and analysis. Routine sample tracking is documented using the Laboratory Sample Analysis Record (LSAR), see [QA-SOP11184](#).

Definitions

1. Laboratory Sample Analysis Record (LSAR): Documentation of the date, time, and analyst for each sample preparation and analysis. The information is compiled in the LIMS using electronic records tracking from the data upload and entry functions. This displays, per sample, on each Analysis Report.
2. Legal Chain of Custody (COC): A hand-signed record showing each change of possession for a sample and/or extract/digest container as it is taken in and out of storage, prepped, analyzed, etc. The employee must sign and date as they receive and relinquish possession. This documentation may be required for litigation or forensic work.
3. Analysis Request/Environmental Services Chain of Custody: Form used to track submission of the sampling containers to the client and receipt of samples from the client to the lab. This may also be referred to as an External COC.
4. Custody: A sample is in custody if it is in any one of the following states:
 - a. In actual physical possession
 - b. In view after being in physical possession
 - c. Locked up so no one can tamper with it
 - d. In a secured area, restricted to authorized personnel (e.g., in the Automated Storage and Retrieval System (ASRS))

Personnel Training and Qualifications

Training for this procedure consists of reading this SOP. Supervisory (or trainer) review of all COC documentation is performed until the trainer is satisfied that proficiency has been achieved. Training of all laboratory personnel is the responsibility of the department manager. Documentation that this training has been completed must be kept in the employee's training record.

Procedure

NOTE: As with all analytical data, it is extremely important that this documentation is filled out completely and accurately with every sample container transfer. Everyone who handles a COC (Legal or External) is responsible to check for documentation compliance to the point of their acquisition. If changes are made to the COC, they must be made in accordance with the ELLE error correction procedure. It is the responsibility of the person who made an error in documentation to correct the error.

A. External COC documentation

1. External COCs document custody from the sample's collection to entry into ELLE's LIMS by Sample Administration (SA). See [QA-SOP11184](#) regarding External COCs.

B. Internal COC Documentation Requirements

1. Every custody change requires a signature, including release to storage and removal from storage. These signatures use the following format:
 - a. Signatures: First initial, full last name, employee number (i.e. S. Good 12345)
 - b. Date: Month/day/year (i.e. 11/20/18)
 - c. Time: military (i.e. 1500)
 - d. Ink: must be indelible ink, black preferred.
2. Custody change can be person to person or person to place, but never place to place. See Figures 2 and 3 for examples of acceptable documentation.
3. Any changes or corrections must be made in accordance with ELLE error correction procedure.
4. **The person transferring custody must ensure that the complete line of custody transfer is documented.** Disciplinary action may be taken for employees who fail to comply with these important requirements.

C. Master List and Original Sample COCs

1. At the time of sample entry, SA personnel generate a Master List of Chains of Custody (Master List) [S-SA-FRM10731](#) for each sample group.
2. A Secure Storage Chain of Custody Original Sample (Original Sample COC) [Q-EQA-FRM6874](#) is generated by each technical department and bottle type in the sample group (i.e. 3 VOC vials get one COC, while 2 HCl preserved bottles get 2 COCs for analyses in Departments 4027 and 4029).

NOTE: If samples need to be transferred before ELLE numbers are assigned, SA must generate an Original Sample COC as completely as possible using the client sample name.

3. The samples are temporarily stored in a secure location that is named SA HOLD. The transfer of custody to SA HOLD is documented on each Original Sample COC.

Example: Release of original samples into SA HOLD on [Q-EQA-FRM6874](#)

Sample Number(s) in Custody	Released By	Received By	Date of Transfer	Time of Transfer	Reason for Change of Custody	Dist., Extr., or Digest Chain Created (X)
1234567-70	S. Good 12345	SA HOLD	1/2/18	1300	Sample Storage	

4. Each Original Sample COC generated must be documented on the Master List.

Example: Documentation of Original Sample COCs on [Q-EQA-FRM10731](#)

Original Sample Chains		
Bottle Type	Started By	Date Started
1000-mL glass w/HCl (06a)	S. Good 12345	1/2/18

1000-mL glass w/HCl (06b)	S. Good 12345	1/2/18
40-mL glass vial w/HCl/Asc (39)	S. Good 12345	1/2/18

5. Samples are moved from SA HOLD to ASRS. This transfer is documented on the Original Sample COC.

Example: Transfer to ASRS on [Q-EQA-FRM6874](#)

1234567-70	SA HOLD	S. Good 12345	1/2/18	1400	Sample transfer	
1234567-70	S. Good 12345	ASRS	1/2/18	1405	Sample Storage	

6. Movement of the sample within ASRS is tracked electronically via bar codes and a validated LIMS tracking system.

D. Sample Custody in Technical Groups

1. When an analyst takes custody of samples, they must completely and accurately fill out the information requested on the Original Sample COC for each sample bottle. This can occur in several ways:
 - a. The analyst takes custody directly from SA personnel.
 - b. The analyst takes custody via requisition through the ASRS. See [QA-SOP11182](#).
 - c. The analyst takes custody from a secure storage location.
2. Each analyst must accurately document each specific test (analysis) for the sample numbers on the Original Sample COC under the 'Reason for Change of Custody'.
 - a. If additional containers of the sample are created (e.g., subsamples, extracts, distillates, leachates, digests, etc.), then an additional COC form must be created by the department if they do not document this information on the original COC form. [Q-EQA-FRM6862](#) for Metals, [Q-EQA-FRM6854](#) for Leachates, [Q-EQA-FRM6864](#) for Water Quality, and [Q-EQA-FRM6868](#) for subsamples are examples.
 - b. If a department specific COC is created, the analyst must mark an 'X' in the column specifying that a subsample was created on the Original Sample COC.
 - c. All changes of custody involving new containers in the department (e.g., analysis, storage, vials on instruments, etc.) must be documented on a departmental specific COC form or on the Original Sample COC.
3. **The Original Sample COC must be signed by both parties each time the sample bottle physically changes hands.**
 - a. Documentation is required for all shift changes.
 - b. If a sample container is needed for analysis in another department, the Original Sample COC is released to the analyst from the other department, and must then be either returned to the first department or to the ASRS.

4. Legal COC samples stored outside of the ASRS must be stored in a secured area. When samples are entered into this area, the "Received By" column is noted as "Department XX storage." When samples are taken from a departmental storage area, the "Released By" column of the COC is documented as "Department XX storage" (Figure 3).
5. Analysts in possession of samples must return the parent samples and Original Sample COC to ASRS with a minimum of delay.
6. All chains must end with a note of either "All Sample Consumed," "Discard," or "Storage" for the final reason of transfer.

E. Completion of the process

1. After sample analysis, Legal COC samples must be returned, with the Original Sample COCs to the Sample Support Group as soon as possible.
2. All samples must be either discarded or stored in the ASRS as their final storage location, and this must be indicated on the Original Sample COCs appropriately.
3. The Original Sample COCs are retained in files within Sample Support until the Data Deliverables Group personnel retrieves the forms so a copy can be included in the data package.
4. The Data Deliverables Group also retrieves all departmental created COC forms so a copy can be included in the data package. The original copy of all of these COC forms are retained on file by the laboratory.

NOTE: for the Data Deliverables Group personnel who collect COC forms for data packages: If you find a completed COC form that does not get a data package, send the COC form to the project manager for that account. The project manager will determine whether copies of the COCs are to be sent to the client with the reports.

5. Any errors or omissions in COC documentation that cause noncompliances must be noted in the case narrative of the sample data package.

[Q-EQA-FRM6854 Secure Storage Chain of Custody Leachates](#)
[Q-EQA-FRM6862 Department Storage Chain of Custody Metals](#)
[Q-EQA-FRM6864 Department Storage Chain of Custody](#)
[Q-EQA-FRM6865 Secure Storage Chain of Custody Supplemental Information](#)
[Q-EQA-FRM6868 Secure Storage Chain of Custody Subsample](#)
[Q-EQA-FRM6874 Secure Storage Chain of Custody Original Sample](#)
[QA-SOP11182 Sample Requisition](#)
[QA-SOP11184 Laboratory Sample Analysis Record \(LSAR\) Documentation](#)
[S-SA-FRM10722 Environmental Sample Administration Receipt Documentation Log](#)
[S-SA-FRM10731 Master List of Chains of Custody](#)
[S-SS-FRM10672 Chain-of-Custody Transfer Record](#)

Attachment:
[Figure 1 - Example COC](#)



Figure 2 - Example Internal COC Secure Storage

Figure 3 - Example Department Internal COC

End of document

Version history

Version	Approval	Revision information
2	06.JUL.2015	
3	01.MAR.2019	

	Always check on-line for validity. <p style="text-align: center;">Environmental Sample Entry</p>	Level: Work Instruction 
Document number: S-SA-WI10723		Organisation level: 5-Sub-BU
Old Reference: 1-P-QM-PRO-9015502; DOD - SOP-SA-101 Version: 14		Responsible: 5_EUUSLA_Sample Administration_Manager
Approved by: POK9 Effective Date 18-FEB-2020	Document users: 6_EUUSLA_Sample Administration_Sample Entry	

[Revision Log](#)
[Reference](#)
[Cross Reference](#)
[Purpose](#)
[Scope](#)
[Personnel Training and Qualification Procedure](#)

Revision Log

Revision:	13	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure	Reflect current practices	Add the requirement to print a copy of the hold report and attach it to the samples	
Procedure	Reflect current practices	Add a new step to the process where new sample groups will go onto a shelf and be evaluated each morning during the daily PM meeting	
Procedure	Reflect current practices	Added requirement that groups that are rush or collected more than 3 days from receipt must not be put on hold but resolved with CSR immediately.	

Revision:	12	Effective Date:	April 13, 2018
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	

Revision: 12	Effective Date: April 13, 2018
Throughout Document	Reflects re-identification of documents in D4 Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with D4 numbers
Procedure 3	Reflect current procedures Added section about what to do with samples received for another Eurofins company on Lancaster, PA campus

Reference

Not applicable

Cross Reference

Document	Document Title
S-SA-WI10725	Environmental Sample Receipt and Unpacking
S-SA-WI10743	Taking the Temperature of Environmental Samples Upon Arrival at the Lab
S-SA-FRM29480	Air Canister Handling

Purpose

The purpose of this SOP is to briefly describe the procedures used in environmental sample entry. Immediately following receipt at the laboratory, samples are recorded in the Eurofins Lancaster Laboratories Environmental (ELLE) LIMS sample login system. This is one of the most important processes in the operation of the laboratory. The information entered into the computer establishes the foundation of information utilized throughout the laboratory for scheduling, accounting, billing, reporting, marketing, analysis, storage, and quality assurance. Because so many areas are influenced by the information recorded at entry, the importance of this process is evident.

Scope

This SOP describes the general procedures used in computer entry, called sample log-in.

Personnel Training and Qualification

All environmental sample entry personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

Training of all new environmental sample entry personnel is performed during the first few months of employment. This training includes in-depth instruction of all analyses performed by each environmental technical center, holding times, collection requirements, etc. Entries are reviewed to ensure comprehension until the employee is able to demonstrate a clear understanding of requirements.

Procedure

1. While handling samples in SR, all personnel should wear lab coats and safety glasses. Samples are unpacked and inspected in the receipt area. At this time the samples are examined for breakage, agreement with the associated client paperwork, and the temperature of the samples upon receipt is recorded. See [S-SA-WI10725](#) for more information on sample receipt and unpacking and [S-SA-WI10743](#) on taking the temperature of samples.
2. Following receipt, the samples advance to Sample Registration (SR). The individual carts of environmental samples are surveyed by the entry staff for prioritization of entry based on client requested turnaround time and analysis holding time. The sample groups are immediately entered by SR entry staff or placed within storage at the appropriate temperature condition (refrigeration, freezer, or room temperature) until entry. The administrator reads the client's request for analyses and selects the appropriate analysis number for each test requested. The correct account, sample type (matrix), copy routine (reporting), priority, and turnaround time are selected.
3. If it is determined that the submittal group is not for ELLE but is for another Eurofins company on the Lancaster, PA campus, the entry staff will scan all the accompanying paperwork and attach it in an email to group email box, !US19_SA_Lancaster. The sample receiving areas of all companies on the Lancaster, PA campus are part of this group email address. Once it is identified who the samples belong to, they will be delivered or picked up by the appropriate staff.
4. If the sample administrator determines that the samples cannot be entered into the sample entry system due to discrepancies, unclear analyses, or the client requests that the samples be held, then the samples must be entered into the SA Hold Sample program. The program assigns a unique hold number to each held group. The samples are labeled with the assigned hold number and stored in Sample Registration at the appropriate temperature conditions. Samples that need refrigerated will go in the SA walk-in, samples that need frozen will go into the walk-in freezer in the basement, and samples that that can be kept at room temperature will go onto the rack where the air samples are stored. A copy of the hold form should be printed and attached to the samples/cart. Additionally, copies of the hold reports are immediately sent to the assigned client service representative (CSR) and the original client paperwork and hold report are filed within SR. New groups that are put on hold will go onto a temporary hold shelf labeled "New Hold Groups". This shelf is located in the SA walk-in. Groups added to this shelf will be reviewed each morning during the SA performance management meeting. If the questions can not be resolved within 1 business day those groups will be moved to the back of the walk-in with all other hold sample groups. A daily e-mail report is sent to all CSRs listing all outstanding held samples that are waiting resolution. The held sample group is not removed from the daily e-mailed report until the completed hold report is returned to SR with written instructions on how to proceed with entry. This hold report is then filed with all the entry paper work. If samples are rush or were collected more than 3 days from receipt the login staff must immediately attempt to contact the CSR (using the CSR phone list) and resolve the issue rather than putting the samples on hold.
5. The compiled information is then typed into the sample entry program.
6. Immediately following computer entry, a working copy of the acknowledgment describing the account to bill and client purchase order number, reporting information, the number of samples and types (matrixes), analyses ordered for each sample, sample collection information, and number of containers for each sample is printed. The number of containers for each sample is represented by the bottle codes entered. This copy of the acknowledgment represents the information entered into the computer and is attached to the client paperwork for auditing, along with the Receipt Documentation Log. The client's account number, group number and the sample numbers are written on the client's paperwork. Labels are printed for each bottle/package in the sample group. These labels are used to identify each sample and bottle/package while in the computer system. A specific label is attached to each sample bottle/package by comparing

the sample ID on the client's label against the sample ID on the ELLE label and confirming the appropriate bottle code for each container. Each label contains a sort code that is used to assist the login personnel to ensure that the container is sent to the correct location (e.g. metal splitting, pH check, soil splitting, etc.) The table used to populate the sort code information is stored in Parallax and maintained by Dept. 6042 management.

7. The sample proceeds to sample preservation and sample storage or to the appropriate technical center. If samples remain in SR prior to delivery to sample storage or the appropriate technical center, the samples are stored within SR at the appropriate temperature conditions for each sample matrix (refrigeration, freezer, or room temperature).

8. If samples must proceed directly to the department without lab labels due to extreme rush or holding time issues, we track them as follows. The containers which need to go directly to the department are checked to make sure they contain all the pertinent information such as the account name, sample ID, collection date and time, and the name and analysis number of the test to be performed. The individual departments record these samples in their lab notebooks by the client name, sample ID, and collection information as necessary. As soon as the lab labels are ready, the labels are delivered to the appropriate department and applied to the appropriate containers. The department then add the laboratory assigned sample number to their notebooks for complete documentation.

9. A Sample Label Audit Notification form is generated for every tenth entry group. The individual sample labels for that group are audited for accuracy by someone other than the entry person. The auditor is performing a comparison check between the information the client supplied on their label versus what the entry person entered onto the lab label. The auditor initials, dates, and times the audit sheet and indicates whether all labels were accurate. If all labels were not accurate, a description of corrections required is written at the bottom of the form. Whenever possible, the original entry person is responsible for making any necessary corrections to the sample labels, as well as to the acknowledgment. If the original entry person is not available, then another entry person may make the changes and document the changes. All audit sheets are filed by date of entry within SR.

10. The complete entry is audited for correctness in SR. During on the job training all groups are double-checked for accuracy until a complete understanding of requirements are demonstrated.

11. The CSR assigned to the account reviews the entry for completeness and correctness.

12. If the computer entry for a sample must be corrected or changed in any way, a change form is electronically generated to document the changes made and to communicate the change to the impacted departments. The change form identifies what changes were made, which samples are affected, why the change was necessary, who made the change and when. The change forms are automatically emailed to the person who made the change, the CSR assigned to that account and to the contacts in each of the technical centers affected by the changes.

13. When all audits and changes are completed, the hard copy paperwork is filed in Client Services.

[S-SA-FRM29480 Air Canister Handling](#)



[S-SA-WI10725 Environmental Sample Receipt and Unpacking](#)

[S-SA-WI10743 Taking the Temperature of Environmental Samples Upon Arrival at the Lab](#)

End of document

Version history

Version	Approval	Revision information
12	30.MAR.2018	
13	23.OCT.2018	
14	04.FEB.2020	

	Always check on-line for validity. <h2 style="text-align: center; color: red;">Environmental Sample Receipt and Unpacking</h2>	Level: Work Instruction 
Document number: S-SA-WI10725		Organisation level: 5-Sub-BU
Old Reference: 1-P-QM-PRO-9015504; DOD - SOP-SA-103 Version: 18.1		Responsible: 5_EUUSLA_Sample Administration_Manager
Approved by: X6TJ Effective Date 28-JUN-2019	Document users: 5_EUUSLA_Sample Administration_Manager, 6_EUUSLA_Sample Administration_Sample Unpacking	

Note: Handling of Quarantine Soils approval is through Quarantine Soil Permit P525-190620-001 - Robert Dempsey (ELLE President), permittee

- [Revision Log](#)
- [Cross Reference](#)
- [Purpose](#)
- [Scope](#)
- [Personnel Training and Qualification](#)
- [Procedure](#)
- [A. Documentation of Receipt](#)
- [B. Unpacking procedures](#)
- [C. Placement of samples onto carts for entry](#)
- [D. Unpacking problems/paperwork discrepancies](#)
- [E. Temperature of coolers](#)
- [F. Safety procedures](#)
- [G. Cleaning of Coolers and Kits](#)

Revision Log

Revision:	18	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Personnel Training and Qualification	EHS requirement	SR personnel must attend Environmental Sample Hazard Communication Training	
Procedure B.	Performed in another department EHS requirement	Removed headspace check in TB. Added review of USDA Quarantine Soil Permits	
Procedure D.	Clarification	Reorganized procedure for clarity. Added that gross headspace issues must be communicated to CSRs.	

Procedure F.	EHS requirement	Added required waste stream for discard. Spills must be called into 1 1 1
Procedure G.	EHS requirement	Disinfectant used is Vesphene. Added when to contact EHS team.

Revision:	17	Effective Date:	02-NOV-2018
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure	Reflect current practices	Replaced references to SA (Sample Administration) with SR (Sample Registration)	
Procedure A	Reflect current practices	Clarified who needs to write the returned time on the driver pickup list	
Procedure B	Reflect current practices	Added reference to verifying that each container is properly identified before removing from bags.	
Procedure C	Reflect current practices	Added reference use of numbered table markers on carts	
Procedure D	Reflect current practices	Clarified how client services was notified of problems with incoming samples	
Procedure F	Reflect current practices	Changed "bad smell" to "strong odor"	
Procedure G	Reflect current practices	Added additional information on discarding damaged or contaminated coolers	

Cross Reference

Document	Document Title
M-FRM12743	Shipping Request
S-SA-WI10743	Taking the Temperature of Environmental Samples Upon Arrival at the Lab
QA-SOP11893	Environmental Hazardous Sample Communication Procedure

Purpose

Receipt of samples and documentation of receipt are very critical steps in the overall processing of samples. It is very important that proper handling procedures are established and closely followed to ensure the integrity of the sample is maintained and clients' needs are met. It is the

responsibility of the Sample Receiving personnel to properly unpack, document, and communicate information about the samples to the Sample Registration (SR) personnel. All steps must be documented and traceable.

Scope

This SOP is designed to cover all procedures involved in proper receipt of and unpacking of environmental samples in SR.

Personnel Training and Qualification

All SR entry and sample receipt personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP, [S-SA-WI10743](#) and [QA-SOP11893](#). All SR entry and sample receipt personnel performing this procedure must attend Environmental Sample Hazard Communication Training prior to handling any samples.

Procedure

A. Documentation of Receipt

All samples received via courier (i.e., FedEx, UPS, U.S. Mail, laboratory courier, etc.) must be documented on the Sample Administration Receipt Documentation Log (Doc Log). The Doc Log is generated through the Doc Log program in our LIMS system. All information must be accurate, clear, and complete.

Samples received via ELLE's (Eurofins Lancaster Laboratories Environmental) Transportation (samples picked up by ELLE's couriers) are also documented on the Transportation Pickup schedules. As each lab courier returns, the receipt person and the courier must verbally and visually verify that each sample group is accounted for as listed on their pickup list. The courier must write the time that they returned to the lab at the top of his/her corresponding sheets.

The chain of custody (COC) accompanying all samples must be signed in the "Received for ELLE by" space by the receipt person, or the last "Received by" portion of the chain.

B. Unpacking procedures

Begin by looking for the expected RUSH/SHORT HOLD samples for that day. Unpack those first using the procedures outlined below.

1. Initiate the Doc Log for the environmental samples to be unpacked. A separate form must be generated for each submittal group of samples.
2. A unique Doc Log ID number is generated as each new doc log is initiated. This ID is entered into the LIMS system during the sample entry process to associate each entry group with its corresponding doc log.
3. The unpacker completes the form by identifying the client/project name, date of receipt, time of receipt, delivery method, number of packages, and state of origin.
4. The next section of the Doc Log is the "Arrival Condition Summary" where the following conditions are documented with a "Yes" or "No" answer:
 - a. Is the shipping container sealed?

- b. Is a custody seal present?
- c. Is the seal intact?
- d. Is the package chilled?
- e. Is paperwork enclosed?
- f. Are the samples intact?
- g. Are any samples missing?
- h. Are any extra samples present?
- i. Are there discrepancies in container quantity and type of container from the COC?
- j. Do sample ID's on COC match containers?
- k. Do sample date/times match COC?
- l. If VOA vials are received, is there a trip blank included?
- m. Are any of the sample air? If so,
 - i. Were flow controllers received?
 - ii. How many?
 - iii. Were any empty canisters returned?
 - iv. If so what are the corresponding ID numbers of those canisters?

5. Samples received in our kits, coolers, or client packaging are opened and visually checked for shipping damage. If damage is encountered, follow procedures outlined in the Unpacking problems/paperwork discrepancy, Section D.

6. Once visual assessment is complete, remove all packaging material and place it on the unpacking table.

7. Initially, remove all large sample containers from the cooler and place them on a cart or on the table, leaving all the 40 ml volatile vial containers in the cooler with the ice.

8. Prior to unwrapping or removing sample containers from bubble wrap or baggies, verify that the individual containers are labeled with the sample identification and that the identification was not on the outside of the packaging only. If the identification is only on the outside of the packaging, keep the containers in the bag, do not remove them.

9. Next, read the client COC and/or other accompanying paperwork and verify that all contents have been received, reconciling the larger

containers first.

10. If you encounter missing sample(s), broken samples, or sample/paperwork discrepancies, follow the Unpacking problems/paperwork discrepancy procedures, Section D.

11. Once the larger containers have been reconciled against the client paperwork, remove the 40 ml volatile vials from the cooler and reconcile those containers.

12. Remove all wet ice and ice packs from shipping container. The wet ice must be placed in the ice disposal tank. If client paperwork is not included, retain some portion of the packaging for identification (i.e., shipping label, FedEx slips, etc.)

13. For samples received by ELLE's drivers,

a. Begin by scanning the drivers' pickup schedule for RUSH, microbiological, or other short holding time analysis samples. Unpack those samples first. To quickly retrieve those or any other samples, look for the cooler number indicated on the pickup schedules and go to the corresponding cooler.

b. If the driver indicated that paperwork was included, make sure that you have that paperwork.

c. Check samples received vs. the client paperwork. Again, if a discrepancy exists, follow the Unpacking problems/paperwork discrepancy procedures.

d. If a discrepancy is found, check with the driver to see if he/she has any information or can help resolve the current problem or any similar problems in the future.

e. Initial the driver schedule next to the "unpacker" space if, and only if, you actually unpacked that group of samples.

f. At the end of the day photocopy all the drivers' schedules and return the originals to the Transportation supervisor.

14. Once the samples have been removed from the shipping containers and matched against the client paperwork and all required USDA Quarantine Soils permits (with any discrepancies properly documented), the unpacker places his/her signature in the final received by section of any client-submitted COC with the date and time of receipt at the laboratory.

15. The unpacker's name and their employee number will be recorded at the bottom of the Arrival Condition Summary section of the Doc Log, followed by the date and actual time of sample unpacking. The unpacker will print the Doc Log once all the information pertaining to that submittal group has been recorded and give the Doc Log and client's paperwork to the entry staff.

C. Placement of samples onto carts for entry

1. All groups are to be placed on carts.

2. Multiple groups may be placed on the same cart as long as the groups are kept distinctly separate from other clients' sample groups.

3. All samples are lined up according to the sample order on the client paperwork, or if none exists, in the most obvious manner.

4. All glass vials must be put in foam holders and placed with the other bottles from the same sampling point.
5. Place one of the numbered table markers with the group on the cart and write the corresponding marker number on the back of the client's COC.
6. Print the doc log and take the client paperwork along with the doc log to the group leaders in the sample entry room.
7. When each cart is full, or 30 minutes since samples were first placed on carts, or all samples are unpacked (whichever would come first), deliver the cart of samples to the SR personnel.
8. If for some reason all SR personnel are unavailable, the carts of samples are placed within storage at the appropriate temperature conditions similar to how the samples were received (i.e., refrigerated, frozen, or room temperature).

D. Unpacking problems/paperwork discrepancies

The SR person must immediately communicate all problems that will affect the analysis to the appropriate Client Services Representative (CSR) via email or phone call.

Copies of the Doc Log and any client paperwork must be given to the CSR. Client Services must call the client and document the resolution. All Doc Logs are filed with the first page of the client acknowledgment and client paperwork.

Below are procedures for possible unpacking problems or discrepancies:

1. Missing Samples

- a. In the case of missing sample(s), the receipt person unpacking the samples must notify a SR person immediately. The SR person must also verify that the sample(s) are missing by completely checking the shipping container, the packaging contents, and comparing the paperwork and the received containers.
- b. Once the SR person and the unpacker are satisfied that the sample(s) are missing, the unpacker must begin documentation in the Doc Log program. By answering "Yes" to whether samples were missing, the "Details Editor" section of the program will open where the specific missing sample ID's and comments can be recorded.

2. Broken Samples

- a. Broken samples must be removed from the shipping container and disposed of appropriately. In some cases the sample may be salvageable (i.e., a soil in a cracked container can be transferred to a new jar, etc.). The unpacker must judge this situation and if in question check with a SR person. Any information on completely broken sample containers or partially broken/transferred containers must be documented on the Doc Log. By answering "No" to whether samples are intact, the "Details Editor" section of the Doc Log will open where the specific sample IDs and comments can be recorded. Broken or severely damaged shipments may be photographed with a camera, if it is felt that we need to document sample condition. This is an optional piece of documentation, not required in all circumstances.

3. Additional Discrepancies and Problems

The following must also be documented in the "Details Editor" section of the doc log. Any specific sample IDs and information on the containers which differs from the COC are recorded.

- a. Sample labeling vs. paperwork identification
- b. Container quantity discrepancies
- c. Date/time discrepancies
- d. Gross headspace issues (i.e. every vial contains visible headspace)

E. Temperature of coolers

For all environmental samples, the temperature upon receipt is required documentation. See S- SA-WI10743 (Taking the Temperature of Environmental Samples Upon Arrival at the Lab).

F. Safety procedures

The following safety procedures must always be followed during the sample receipt process:

1. Latex or nitrile gloves must always be worn and replaced if torn or soiled.
2. Thermal gloves are available for use when handling samples received in dry ice. In addition, cut-resistant gloves are available for use when handling loose ice containing broken glass.
3. Lab coats and safety glasses must always be worn.
4. If a sample meets any of the following conditions, it must be placed in the hood:
 - a. Strong odor
 - b. Expanded, bulging containers or septums
 - c. Fuels (neat)
 - d. Labeled as hazardous
 - e. Breakage and/or leakage

NOTE: Any samples labeled as or materials contaminated with hazardous quarantine soils must be disposed of through the EHS waste stream.

(If ever in doubt, place in the hood until a technical decision can be made.)

5. Where warranted appropriate warning stickers must be used (i.e., Hazardous, Quarantined Soil). Guidelines for identifying and communicating information about potentially hazardous samples are in [QA-SOP11893](#).

6. Any spills or releases of material must be called into 1 1 1 for ERT response.

G. Cleaning of Coolers and Kits

1. ELLE coolers and kits

All coolers/kits must be completely cleaned after all the samples are unpacked with Vesphene (anti-microbial disinfectant).

- a. All packaging material must be discarded properly.
- b. All shipping tape and labels removed from the inside and outside of the cooler/kit, with the exception of the ELLE identification tag.
- c. The cooler/kit must be completely wiped out so there is no remaining water in the cooler/kit.
- d. The interior of the cooler/kit must be wiped down with Vesphene to remove any residual moisture and visible debris so the cooler is reasonably clean and ready for future use.
- e. The cooler/kits must be returned to the bottles department for future use.
- f. In the event that a cooler/kit is damaged or contaminated it must be discarded and not used any further. To do so, place the cooler in the area outside of the unpacking room designated for "Coolers for the Dumpster". Any coolers contaminated with hazardous quarantine soils must be segregated and the EHS waste team contacted for proper disposal.
- g. Coolers that need to be discarded are taken to a designated spot out in the hallway where facility services will dispose of them.

2. Client coolers and kits

All client coolers/kits must be completely cleaned following the procedures above with the following exceptions:

- a. Any temperature blanks, ice packs, and/or special packaging material supplied by the client will be returned in their cooler to them if they supplied a return shipping label or have supplied the lab with their courier account number.
- b. Form [M-FRM12743](#) must be completed for return to the client with the client's commercial courier billing account number filled in on the form.
- c. The coolers/kits are taken to the Shipping Department with the Shipping form taped to the lid of the corresponding cooler/kit.

[M-FRM12743 Shipping Request](#)



[QA-SOP11893 Environmental Hazardous Sample Communication Procedure](#)

[S-SA-WI10743 Taking the Temperature of Environmental Samples Upon Arrival at the Lab](#)

End of document

Version history

Version	Approval	Revision information
17	30.OCT.2018	
18	21.JUN.2019	
18.1	28.JUN.2019	Editorial only, added Quarantine Soil Permit info

	Always check on-line for validity. Taking the Temperature of Environmental Samples Upon Arrival at the Lab	Level: 
Document number: S-SA-WI10743		Work Instruction
Old Reference: 1-P-QM-PRO-9015508; DOD - SOP-SA-138		
Version: 16		Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date 18-NOV-2019	Document users: 5_EUUSLA_Sample Administration_Manager, 6_EUUSLA_Sample Administration_Sample Unpacking	Responsible: 5_EUUSLA_Sample Administration_Manager

[Revision Log](#)
[Cross Reference](#)
[Purpose](#)
[Scope](#)
[Personnel Training and Qualification](#)
[Procedure](#)

Revision Log

Revision: 16	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Procedure A. 2-6	Enhancements	Revised procedure for taking temperatures during triage using new form S-SA-FRM28783

Revision: 15	Effective Date:	17-July-2019
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Purpose	Enhancement	Added temperature requirement 0-6 C
Scope	Enhancement	Added that agency and client specific procedures are followed as needed
Procedure A.3.	Regulatory Requirement	Added when temp bottles are used. Surface temps are taken for courier and drop off samples.
Procedure F.7.	Enhancement	Revised process for out of spec temperatures

Cross Reference

Document	Document Title
S-SA-FRM10729	Infrared Thermometer Check Log
S-SA-WI10725	Environmental Sample Receipt and Unpacking
S-SA-FRM28783	Triage Sheet
S-SA-FRM10719	Sample Receipt Documentation Log Continuation Page

Purpose

The purpose of this SOP is to instruct Sample Receipt personnel and trained backups how and when to take the temperature of environmental samples upon receipt at the lab. It also explains the documentation procedures required. Temperature requirements are between 0 and 6 degree Celsius.

Scope

This SOP is designed to cover all procedures involved in taking and documenting the temperature of environmental samples upon receipt at the lab. If agency or client-specific requirements or QAPPs specify procedures different from our normal procedures, we will follow those requirements.

Personnel Training and Qualification

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and [S-SA-WI10725](#).

Procedure

A. Procedure for taking the temperature

1. Take the temperature of the shipping container upon receipt at the lab for:
 - a. Samples shipped to us in any type of cooler, Styrofoam kit, or insulated box
 - b. Samples brought to the lab by the lab driver
 - c. Samples delivered by the client in any type of cooler, Styrofoam kit, or insulated box
 - d. Samples brought to the lab by lab field samplers
2. The temperature is taken during the triage process which occurs immediately after sample receipt.
3. Since the sample bottles are not removed from the cooler during triage the temperature information is documented on the Triage Sheet [S-SA-FRM28783](#).

4. If the cooler temperature is high the user must also complete Sample Receipt Documentation Log Continuation Page [S-SA-FRM10719](#)
5. Our LIMS system contains a program which creates an electronic version of the Sample Administration Receipt Documentation Log (Doc Log). The Doc Log contains a field to indicate "Yes" or "No" whether the client's samples were received chilled.
6. When the samples are unpacked the technician will use the information from [S-SA-FRM28783](#) to populate the appropriate fields in the electronic Doc Log
7. The two methods used to measure the temperature of samples in a cooler are a "temperature bottle" or a surface temperature of a sample container.
 - a. Temperature bottles are used on samples that were collected on a previous day so the lab is sure that all samples have been in contact with the ice for an appropriate amount of time to properly chill them. This procedure primarily applies to samples received via commercial courier (e.g. FedEx or UPS). It would also apply to samples subcontracted to us by other labs or one of our sister labs.
 - (1) Remove the temperature bottle from the cooler. Close the lid while taking the temperature to keep the samples chilled.
 - (2) Invert the bottle several times to ensure consistent temperature throughout the bottle. Remove the lid and insert the digital thermometer probe into the water.
 - (3) After the temperature reading stabilizes, record the thermometer reading in the Doc Log application.
 - (4) If the temperature bottle is significantly frozen, document this on the Doc Log and use the IR gun to check the surface temperature as described below.
 - b. Surface temperature of the bottle is used when samples are collected on the same day of arrival. This typically applies to samples brought back to the lab by one of our drivers or if they are dropped off by one of our clients. This procedure would also apply to samples collected on a previous day if no temperature bottle is included.
 - (1) One temperature from each sample matrix in the cooler must be taken.
 - (2) If the last sample was collected more than 4 hours prior to the time it is being unpacked any sample container can be used for the temperature check.
 - (3) If the last sample collected was less than 4 hours prior to the time it is being unpacked a container from the last sample collected must be used for the temperature check.
 - (4) Touch the infrared thermometer's nose piece directly onto the label of the client's container.
 - (5) Pull the trigger on the side of the infrared thermometer and wait until the temperature is displayed on the thermometer.
 - (6) Always attempt to take the reading from the white paper adhesive label on the container. This will provide a fairly consistent surface from which to take the reading.

(7) Try to avoid ever taking the reading from a metallic or shiny surface. If the shipping container only contains glass vials, take the temperature from the flat bottom of the vial and not the rounded side.

B. Procedure for documentation of temperature by unpacking personnel

1. In the "Samples Chilled" section of the Doc Log, select the correct thermometer ID (located on each thermometer), read the temperature from the thermometer and enter this temperature into the Doc Log. Then hit the field "apply correction".

2. The following items must also be recorded on the Doc Log:

- a. Thermometer ID
- b. Method of taking the temperature digital (temperature bottle) or infrared (surface temperature)
- c. Whether Wet Ice/Dry Ice/Ice Packs were used as the cooling device
- d. Whether ice was still present when wet or dry ice was used
- e. Whether the ice was loose or bagged within the shipping container

3. If specific packing procedures are observed which may have contributed to an elevated temperature, note these in the conditions contributing to elevated temperature section of the Doc Log.

C. Denoting multiple temperatures per sample group

In some cases the sample containers for a particular sampling location or sampling group are packed in multiple coolers. It must be made clear which sampling containers were at which temperatures. The Samples Chilled section of the Doc Log can expand to record the individual temperature of each shipping container submitted by the client. If an individual chain-of-custody form exists for each shipping container, the number of the temperature slot associated with that cooler can be referenced at the top of the corresponding COC.

D. Thermometer calibration checks

The infrared thermometer and the digital thermometers must be calibrated quarterly. However, users must check at the beginning of each day to confirm that the units are still within the calibration window.

E. Documentation of the temperature on the client paperwork/chain of custody

The person unpacking the samples must document the temperature on the client's paperwork or chain of custody. If there are multiple temperatures per group, the documentation will show the range of temperatures for the entire sample submittal group.

F. Out-of-range temperature procedures

1. For samples received via ELLE driver or dropped off by a client, there are no specified temperature acceptance limits as long as the samples were submitted the same day as collection and with ice present in the cooler/s. (NOTE: This allowance for same day receipt with ice is not acceptable for samples from West Virginia.)

2. Clients must be notified of any temperatures out of this range so that they may make adjustments to their packing procedures in the future. If agency or client-specific requirements or QAPPs specify procedures different from our normal procedures, we will follow those requirements.

3. If the initial temperature recorded from the temperature bottle or surface temperature is above 6°C, the program will flag the unpacker that the cooler is elevated.

4. Go to the "Elevated Temp" section of the Doc Log and determine if the samples were collected the same day as receipt and if they were submitted with wet ice.

5. If so then flag them on the Doc Log with "samples collected same day as receipt".

6. If there are less than 5 containers only the temperature of the containers in the cooler will be taken.

7. If not collected same day, spot-check samples from the four corners and center of the shipping container using the IR thermometer.

a. Temp Bottle

1. If all of the containers are between 0 and 6 degrees then these are considered to be within an acceptable temperature and we can proceed with the analysis.

2. If all those temperature readings are above 6°C, record the 6 individual temperatures from that package (initial, four corners, and center) and note it on the Doc Log

b. Surface Temp

1. Since the initial temp reading was from a container in the cooler do not retake a temp in the section of the container that the bottle was in.

2. Document the initial temp reading in the section where it was placed within the cooler in addition to the 4 other temp readings

3. If any of those subsequent temperature readings are above 6 degrees, continue to check the surface temperature of every sample container within the shipping container and identify those which were received within the proper temp range.

8. Record the sample ID's of all the samples contained in that elevated cooler on the Doc Log.

9. The Doc Log for each submittal group is attached to the entry group's acknowledgment, along with any client paperwork, which had been submitted with the samples.

10. This packet of information is reviewed each day by the assigned client service representative as part of the audit process. After auditing, it is filed numerically by account number in SA's files.

G. Client submitted temperature monitoring devices

If the shipping container contains a client-submitted temperature monitoring device, we must follow the device's instructions for stopping the temperature recording process. The date and time that the device was stopped must be recorded on the Doc Log. Place the device on the cart with the samples and send through to the entry area to be returned to the client as instructed. We must still follow our routine procedures for taking the temperature of the shipping container and document that on the Doc Log.

H. State specific requirements

State specific temperature requirements are incorporated into the electronic Doc Log program whenever possible. For instance, when samples are submitted from the state of WV, the program requires the unpacker to take the temperature of every container in the cooler and records the temperature range within each cooler.

[S-SA-FRM10719 Sample Receipt Documentation Log Continuation Page](#)

[S-SA-FRM10729 Infrared Thermometer Check Log](#)

[S-SA-FRM28783 Triage Sheet](#)

[S-SA-WI10725 Environmental Sample Receipt and Unpacking](#)

End of document

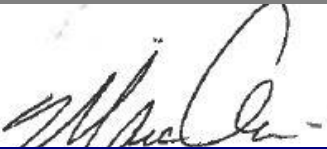

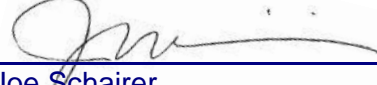

Version history

Version	Approval	Revision information
14	15.JUL.2016	
15	11.JUL.2019	
16	04.NOV.2019	

Appendix G

Waste Disposal

Title: Waste Disposal

Approvals (Signature/Date):	
 _____ Marc Onishi Technical Manager	12/03/2018 Date
 _____ Lisa Stafford Quality Assurance Manager	11/29/2018 Date
 _____ Joe Schairer Health & Safety Manager / Coordinator	11/30/2018 Date
 _____ Chris Williams Laboratory Director	11/29/2018 Date

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1. PURPOSE

- 1.1. The purpose of this procedure is to provide details for waste management procedures established in Corporate Environmental Health and Safety Manual (CW-E-M-001) Section 13 that apply to TestAmerica Sacramento.

2. SCOPE

- 2.1. TestAmerica Sacramento is a Large Quantity Generator (LQG), which creates over 1000 kg/month of hazardous waste (CW-E-M-001 Section 13.3). Consequently, TestAmerica Sacramento maintains an Emergency Response Team (ERT) for spills.
- 2.2. Drums must be shipped within 90 days from the date waste is first placed in them. Only non-hazardous soapy water from glassware cleaning is disposed via laboratory sinks to the sewer (CW-E-M-001).

3. SAFETY

- 3.1. Procedures shall be carried out in a manner that protects the health and safety of all associates. When moving a waste drum, always use a drum dolly, and a ramp for palletizing. Pipettes, broken glass and VOAs will be collected and transported in cardboard boxes to prevent lacerations. Drums will be closed and liquid removed from the top to prevent spills in transport. Secondary containment will be used in liquid transport where possible to prevent spills. Doors will be kept closed as much as possible to discourage intrusion. Lab trash buckets should be carried to the drum and the tied bag placed inside with the opening facing up to minimize lacerations or spills. OSHA mandates the use of spring loaded vent bungs on solvent drums to prevent pressurization explosions.
- 3.2. The sump in the waste processing area where lab wastewater is collected in a holding tank is a confined space that requires special training equipment and preparation before entry. Contact the EHSC before entering the sump. The West Sacramento Fire Department may be able to provide the needed fall protection/extraction harness.
- 3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor.
- 3.4. In addition to lab coat and protective safety glasses, the following personal protective equipment is required for specific tasks:
 - 3.4.1. When moving 55-gallon drums, the waste processor will wear steel toe boots or toe guards and work gloves.

- 3.4.2. When adding bottles to glass drums, crushing glass and lab trash, or cleaning the Vyleater, puncture resistant gloves are required.
- 3.4.3. When operating either the Rampactor or Vyleater, hearing protection is required.
- 3.4.4. Wear a faceshield when pouring HF, concentrated acids or bases, or solvents.
- 3.5. Pollution Prevention
 - 3.5.1. Drain Protectors (magnetized sheets) are used to cover/close the affected storm drain during load out for waste shipment.
 - 3.5.2. The use of sumps and secondary containment throughout the building helps to minimize the potential for contaminants to escape if they are spilled.
 - 3.5.3. Routine testing and monitoring of POTW discharge ensures that we remain in compliance with regulatory limits.
 - 3.5.4. The presence of specially treated hard surfaces in building sumps and work areas prevents leakage of contaminants into the ground.
 - 3.5.5. The use of closed containers and spring loaded vent bungs minimizes air pollution.
 - 3.5.6. Earthquake cables in H-3's and banding pallets minimizes risk of drums falling over.
- 3.6. Waste Management
 - 3.6.1. Contaminated tools and materials (that cannot be cleaned) become hazardous waste, and must be disposed of as the original contaminant.
 - 3.6.2. Special spill response equipment includes drum lifter lever, and UN rated 85-gallon salvage drum. Leaky drums should be placed with the top towards the top of the salvage drum to facilitate final treatment and disposal.

4. DEFINITIONS

- 4.1. Waste: Material no longer useful in the laboratory requiring disposal.
- 4.2. Drum: Cylindrical container for holding waste. Usually 55-gallon, may range from 5- to 85-gallon.
- 4.3. Satellite container: Smaller container near the point of waste generation that is emptied at least once a year.

- 4.4. H-3 Room: Purpose built room for bulk waste storage. They feature fire doors, explosion proof wiring/lighting, separate ventilation, waxed sprinkler heads, and a grated sump to contain waste spills.

5. PROCEDURE

Wastes are either excess sample or analytical wastes. Analytical waste may include reagents (excess, expired, off-specification, or spilled), expired standards and curves, sample extracts, extracted solid or liquid samples, and machine fluids such as coolant and vacuum pump oil. Samples may accumulate in laboratories (filters in Air Tox, VOAs and core tubes in VOA instrument) or may be returned to Sample Control.

- 5.1. Waste Collection: Waste is collected at multiple locations around the facility, then consolidated at either H-3 Waste Accumulation Rooms or the main waste collection area.
- 5.1.1. Waste is collected in the lab as outlined in Section 5.3 through 5.7. Labels are applied to all containers to identify the type of waste, hazardous constituents, collection start date or last emptied date, and the relative hazard of the waste. The National Fire Protection Association (NFPA) diamond system is our preferred method. Pre-made labels for satellite collection containers are available through EH&S, and are maintained on the safety drive and the public drive, in the one of the “labels” folders.
- 5.1.2. Satellite collection areas and containers are found in all prep and instrument labs. These must be emptied when they are full, or no more than one year after the first waste is put into the container, whichever comes first.
- 5.1.2.1. Instrument satellite waste containers are typically 2.5-liter to 20-liter plastic or plastic-coated glass carboys. When full, these are either dumped into a 55-gallon drum in an H-3 closet or moved directly to the main waste area, depending on the type of waste. Refer to the specific operations SOP.
- 5.1.2.2. Lab satellite trash cans are 5-gallon plastic buckets, with lids. When emptied, these are dumped into a 55-gallon drum in an H-3 closet or the main waste area.
- 5.1.2.3. Glassware satellite collection containers are cardboard boxes that are moved to the main waste area for disposal.
- 5.1.2.4. Storage cabinets in the main waste area.
- 5.1.3. 90-day collection areas are the “H-3” closets located between the metals prep lab and the air toxics prep lab; between sample administration and the organic prep lab; and between the organic prep lab and the low-resolution dioxin prep

lab. There is a 55-gallon drum for incinerable lab trash and a 55-gallon drum for landfill lab trash in general chemistry. In the sample administration area, there is a 55 gallon drum for incinerable lab trash, and a 60 gallon yellow garbage can for AEEE packing materials (to be disposed as incinerable.)

- 5.1.3.1. At the air toxics/metals prep H-3; there are drums for the accumulation of landfill lab trash and acid waste.
- 5.1.3.2. At the sample administration/organic prep H-3, there are drums for the accumulation of Methylene Chloride waste, LLE waste, landfill and high solvent lab trash, and used disposable extraction glass trash.
- 5.1.3.3. At the organic prep/low resolution dioxin prep H-3, there are flammable solvent waste, HPLC waste, high solvent and landfill lab trash, and LLE waste water.

5.2. Sample Disposal Procedure

5.2.1. Create a Container Disposal Report

- 5.2.1.1. In TALS go to Sample Management and click on Internal Chain of Custody.
- 5.2.1.2. Change the Search By box to Current Location
- 5.2.1.3. Click the Current Loc box and select the location you would like to search. Then select OK.

Note: Only one location can be searched at a time.

- 5.2.1.4. Select the Search – Append box on the top far right to load all containers on that shelf.
- 5.2.1.5. Once all shelves have been searched, right click on grid and Select Disposable Containers.
- 5.2.1.6. TALS will ask you if you want to bypass the disposal days in the project, select no.
- 5.2.1.7. TALS will check and highlight the containers ready for disposal (may take a few minutes depending on quantity).
- 5.2.1.8. Right click on the grid and click Clear Unselected (may take a few minutes depending on quantity.).
- 5.2.1.9. Select the Export button on the bottom right of the screen.
- 5.2.1.10. Save the spreadsheet to a location naming it appropriately.

5.2.1.11. Remove any unwanted columns for easy printing (leave the columns titled Container ID, Lab Sample ID, Client Sample ID, Sample Matrix, Container Type, Storage Location and Current Location).

5.2.1.12. Print this list and store in a known location for employees to pull samples for disposal.

Note: These containers have not been disposed of and are only a list of containers ready for disposal.

5.2.2. Remove containers from storage location.

5.2.2.1. The Containers Disposal Report lists the individual containers for disposal, not samples nor entire jobs. Only remove the containers outlined on the disposal report. Ask the Sample Custodian about the status of remnant containers in a job being disposed.

5.2.2.2. Remove the containers to a cart and check them off on the Container Disposal Report. Use “NF” or “0” to designate containers not found. Using only the top shelf of the cart and arranging in neat rows will facilitate scanning out.

5.2.3. Scan the containers into TALS for disposal.

5.2.3.1. Open TALS (menu 40) in your own username and password. Click Sample Management, then select Internal Chain of Custody.

5.2.3.2. Change the Search By box to read Lab Sample ID.

5.2.3.3. Scan every container into TALS.

5.2.3.3.1. Right click on the grid and choose Select Disposable Containers.

5.2.3.3.2. TALS will ask you if you want to Bypass the disposal days in the project. Select ‘no’.

5.2.3.3.3. TALS will generate a list recommending disposal drums for each container. LLE (liquid-liquid extraction water) is the default for containers with the least hazard, regardless of matrix. RCRA Soil is for solids having higher levels of metals or organics. HPLC (high performance liquid chromatography waste) is for aqueous samples with high organic contamination. Acid Drum is for aqueous samples high in metals. PCB (polychlorinated biphenyls) and Dioxins will go to lab packs.

5.3. Liquids: refer to Section 5.14, waste list.

Laboratory analysts are responsible for disposing liquid waste as listed in the specific operations SOPs. These will provide the following direction:

- 5.3.1. Dispose waste liquids to the appropriate container in the lab area.
- 5.3.2. Acid, Liquid-Liquid Extraction (LLE), Solvent, and High Performance Liquid Chromatography (HPLC) wastes:
 - 5.3.2.1. When the satellite collection container is ready to dump, transfer the contents to the appropriate drum in the H-3 Room.
 - 5.3.2.2. When the solvent drum is full (four to six inches from the top), bung the drum and transfer it to the waste collection area in the warehouse. Determine “fullness” with a wooden dowel or dipstick.
 - 5.3.2.3. Return with an empty drum to H-3, attach and date a proper in-house label.

WARNING: When using a drum dolly to move either a full or empty 55-gallon drum, you must always face the drum.

- 5.3.3. Deliver other wastes and expired liquids (VOA vials, standards, curves, and extract vials) directly to the main waste room.

5.4. Processing Liquid wastes

- 5.4.1. VOA liquid waste: The VOA analysts collect analytical waste in 4-L plastic jugs. These are transferred to the processor and treated as follows:
 - 5.4.1.1. Add two tablespoons of sodium bicarbonate (baking soda) to each jug to assure pH 6-8.
 - 5.4.1.2. Pour into a blue plastic LLE waste drum.
- 5.4.2. VOA vials: VOA analysts collect 40 ml vials with remaining unused sample of the purged VOA vial in cardboard boxes lined with plastic bags. Unused and expired aqueous and soil sample are removed from refrigerated storage and collected in VOA boxes. When the collection boxes are full or after no more than one year all waste VOA vials are transferred to the main waste room. Once they are transferred to the main waste room, the processor performs the following:
 - 5.4.2.1. Place boxes of VOA vials on the narrow table next to the blue Vyleater. Check all fingerscrews on the Vyleater for tightness. Power up by throwing the main power switch to ON. Place a 20-L plastic carboy with 250 g of sodium bicarbonate under the Vyleater

with the hose down its throat. Place the rubber mat in front to reduce breakage of fallen VOA vials. Situate a lab trash drum under the right side of the Vyleater. Prepare the drum by powdering the bottom of the drum with sodium bicarbonate to neutralize any hydrochloric acid, followed by a half inch of vermiculite to absorb any liquid.

5.4.2.2. Press the left and right green buttons to start the Vyleater. Pull the adjustment wheel from under the Vyleater and insert just below the hopper. Adjust the rollers to the point where they are almost touching. Load about 10 VOA vials into the hopper and raise it to the top, where it dumps into the Vyleater. Readjust the rollers so that they crush the vials being processed. Pull the adjustment wheel from under the Vyleater and insert below hopper to adjust width between grinder wheels to match vial size. The Vyleater will chew down the vials rapidly when the correct width is achieved. Observe that the liquid flows into the carboy and the broken vials are delivered to the lab trash drum. Use the plastic scoop to add vials to the hopper. This drops fewer on the floor. Repeat process until carboy is full or VOAs are all crushed. Wait until liquid stops flowing before changing carboy. Dump full carboys to the blue plastic drum designated for incineration.

5.4.2.3. When done, clean the Vyleater: Use the sodium bicarbonate squirt bottle to neutralize any HCL in the hopper and grinder area.

WARNING: DO NOT stick any body parts into the Vyleater. Wear heavy rubber coated gloves when cleaning the Vyleater.

Stop the Vyleater by pressing the Red button. Then switch OFF the power on the wall. Open the panel on the right end. Open the inner panel. Use the long handle scraper to clear most of the glass off the top screen. Pull out the screen while brushing remaining glass into the Vyleater. Lean the top screen against the Vyleater front and repeat for the lower screen. Scrape glass on the bottom towards yourself with the scraper. Use the wide putty knife to pick up and dispose to lab trash drum. Replace screens, close Vyleater and remove lab trash drum to Rampactor for more waste.

5.4.3. Process solvent vials in the Vyleater observing the following differences: A The waste solvent into a 4-liter polyethylene jug. The 4-liter jug is then emptied into the flammable waste drum in the Advanced Technology H-3. Five gallon open-top drums delivered by analysts may contain bottles (60-250 mls). These are set aside and poured to the collection drum. Neat materials and standards with hazards other than flammability are set aside for lab packs. Wait overnight for vapors to clear before cleaning the Vyleater.

- 5.4.4. Enter the drum in logbook and in TALS using a sequential number for the year. Document the start date, full date, shipping date and manifest number when shipped. Check the LLE and HPLC drums for pH (6-8 is acceptable).
- 5.4.5. Torque the bungs.
 - 5.4.5.1. For waste packaged in blue poly drums (LLE, HPLC, and Acid), use the yellow preset torque wrench to apply 20 foot-pounds to each bung.
 - 5.4.5.2. For waste packaged in steel drums, (solvents), replace the bungs with a 2 inch self-venting bung to prevent pressure buildup until shipped. Ship with the original bung torqued with a red pre-set torque wrench to 60 foot-pounds.
- 5.4.6. Apply a preprinted paper in-house label and write in drum number and start date. Make sure that the label is applied to upper third of the drum.
- 5.4.7. Next to the waste label, apply a 100 mm square DOT diamond indicating hazard class: 3 and 6 for solvent, 6 for DCM, 8 for Acid, 9 for HPLC drum.
- 5.4.8. Mark the drum on its top and sides with the drum number (from logbook) and profile number (from waste list or logbook). Place the drum number on the side between RCRA and DOT labels, with the side profile number placed immediately below labels. Write the numbers on the top so that they can be read from the label side. Use a black Sanford "Magnum 44" marker on poly drums and the tops of steel drums. Use a yellow Sanford "Mean Streak" marker to mark steel drum sides.
- 5.4.9. Strap drums with the same waste profile together on pallets. When less than four drums are shipped, they may be strapped with other compatible drums going to the same TSDF (Treatment Storage Disposal Facility). Mixed flammables solvent drums are kept in the small shed in the warehouse parking lot until shipment.
- 5.5. Solids: See high VOA lab trash, land fill lab trash, soil and RCRA Soil on waste list, Section 5.14.
 - 5.5.1. Analysts dispose hazardous lab trash to the appropriate container in the lab area (lab trash bucket, glass box).
 - 5.5.2. When the container is full, the analyst transfers it to the waste collection area in the warehouse or to the lab trash drum located in each H-3. Analyst replaces box or liner in lab trash bucket and marks new start date. Lab trash drums in H-3s are transported to main waste room when full, or not more than 90 days from start date.

- 5.5.3. The Processor uses orange Rampactor to consolidate lab trash. Rampacting the waste saves on the number of drums used, transport costs, and brings the drums up to the weight listed on the profile.

WARNING: In addition to lab coat and safety glasses, cut and puncture resistant gloves and earmuffs are required when operating the Rampactor. Before operation, read, follow, and replace the instruction sheets located in the blue capped steel tube welded on the right side below the control levers.

- 5.5.4. Sweep any broken glass out of the Rampactor. Seat drums fully over the bottom metal disk in the chamber. A shard of glass can tilt the drum. When the top disk crushes the edge of the drum, it can become wedged in place.
- 5.5.5. Mark the position for the bottom of the drum. Place a perfect empty open-top drum in the Rampactor chamber. Center it as well as possible over the bottom disk. Close the chamber door and latch. Turn on the electric power switch on the wall to the left of the Rampactor. Push both levers back until the top disk lowers to the level of the drum top. Observe through the tiny slit at the top of the door. Use a flashlight to help see. Pull both handles forward (towards yourself) to stop the ram and disk. Turn off the electric power.
Open the chamber door. Center the top of the drum under the top disk. Draw a circle around the drum bottom on the Rampactor using a yellow Sanford Meanstreak marker. Allow to dry. This allows you to place the drum more accurately. Remove the empty drum
- 5.5.6. Select an open top steel lab trash drum. Drums from lab areas will have a white paper in-house waste label with start date. Use oldest start date data.
- 5.5.7. Log in the drum. Find the drum logbook in the Waste Room bookcase. Turn to the current year and contents page. Turn to the Lab Trash pages. Enter the next drum number in the logbook, on the drum and on the waste label. Mark the profile number on the top of the drum. Use the current date when starting a new drum.

Note: Do not apply start dates from glass boxes. They are satellite containers and may accumulate for one year. Lab trash drums must be shipped within 90 days of the start date.

- 5.5.8. Place the drum in the chamber and center on the disk. Close the chamber door, latching top and bottom. Turn on the electric power. Push both levers away from you and release. Carefully watch the disk descend to make sure it clears the edge of the drum. If it appears that the ram will not clear the edge of the drum properly, jerk back the levers. Then follow the procedures in paragraph 5.5.5 and reposition the drum so that it is centered under the ram and disk. The ram is bottomed when the dial reaches the line at 4 o'clock.

Pull the bottom lever towards you, if it does not pop out automatically. The dial will indicate full pressure again when the ram and disk reach the top. Pull both levers towards yourself. Turn off the electric power. Open the chamber door.

- 5.5.9. Add more bags of lab trash or boxes of glass, and crush until no more can be added, or it is too heavy to move. Mix light plastic with glass to achieve an average weight. Pull bags from drums by the knot. Do not inhale while your head is in or over the drum.
- 5.5.10. Top off the full drum with vermiculite. It is stored in bags under the pallet racks.
- 5.5.11. Put ring and lid on drum. If the drum top is out of round, open the ring further and drop it 1/3 down the drum. Use the Rampactor carefully to seat the lid, then bring up the ring and fasten with a 5/8-inch bolt (from the box under the radio).
- 5.5.12. Torque drum bolt to 60 foot-pounds.
- 5.5.13. Enter "Full" date in logbook. Mark drum top as in Section 5.4.8.
- 5.6. Soil: Bring excess soil samples from Sample Control to the main waste room 30 days after invoice date, so clients have time to review and request additional tests. Soil must be thermally treated to comply with the California and US Department of Agriculture permits to import soils from quarantine areas. Soils with high metals may be drummed under the RCRA soil profile: (CH94287-RCRA)
VOA analysts bring soil samples after testing. Once soil is received, the processor handles the material as follows:
 - 5.6.1. Log, label and mark a drum for soil (profile CH94287). Line the drum with a 55 gallon plastic liner (kept on top of Tank 1 in the pump room).
 - 5.6.2. Place soil samples in the drum. Glass containers and acetate tubes may be placed in whole. Do not open containers marked as high results for lead. Otherwise pour samples of loose, dry consistency over the other containers to fill the space, and reduce the number of waste drums required. Re-cap empty glass containers and place in high VOA lab trash for thermal treatment.
 - 5.6.3. Remove the contents of metal core tubes with a soil sampling gouge furnished with a wooden handle. Reserve the tubes (segregated by metal) for salvage. Soak the core tubes (sleeves) in 10% Chlorine bleach solution to kill agricultural pests.
 - 5.6.4. Attach copies of soil permits for TSDF to soil drum.

5.7. Non Hazardous solids: Empty solvent jugs, and sample bottles.

Analysts drain empty solvent bottles to appropriate waste drum and air overnight in hood to vent vapors. Keep bottles inverted so heavier than air vapors fall out.

In the Organic Prep and Dioxin Prep laboratories, place bottles in a drum and transport to the main waste room. Clean client sample bottles, with identification removed, are collected in the same drum. In other areas, collect and transport empty bottles on tub carts and place into the open-top drums just to the right of the double yellow doors.

Once the empty solvent jugs and sample bottles are received in the main waste room, the processor handles them as described below:

- 5.7.1. Remove empty bottles from the glass collection drum(s) until even with the top of the drum. Otherwise they will fall and splinter. Place a metal lid over the glass to discourage jumpers. Using a drum truck, move the full drum to the Rampactor.
 - 5.7.2. Crush the solvent bottles in the Rampactor and dump the glass into the dumpster using the forklift. The brown glass is borosilicate and melts at a higher temperature than soda ash glass, and is not recycled in California. Glass is crushed the same as lab trash (refer to Section 5.5) with the following exceptions:
 - 5.7.2.1. When client identification can not be removed, sample bottles are crushed with high VOA lab trash to protect client confidentiality.
 - 5.7.3. Place the glass drum in the chamber over the bottom disk. Remove the metal lid. Close the chamber door, latching top and bottom. Turn on the electric power. Push both levers away from you and release. Carefully observe the top disk descend to make sure it clears the edge of the drum. If it does not, jerk back the levers, and adjust as in Section 5.5.5.
 - 5.7.4. The ram is bottomed when the dial stops advancing. Pull the bottom lever towards you, if it does not pop out automatically. The dial will indicate full pressure again when the ram and disk reach the top. If plastic bottles have crawled around the edge, lower the disk a tad to release them. Pull both levers towards yourself. Turn off the electric power. Open the chamber door. Cover the drum to contain dust. Remove the drum or add more glass and repeat. When the crushed glass reaches the chine one-third from the bottom, stop and place the drum in the drum carrier. It is heavy enough.
 - 5.7.5. When the container is one-third full, contents are transferred to a dumpster, and eventually are disposed in the local municipal landfill.
- 5.8. Dumping glass to the dumpster:

- 5.8.1. Open the rollup door in the main waste room all the way up. Position the drum carrier a few feet back from the doorway. There is a berm at the door so the forks will bounce up and down as your front forklift tires go over.
- 5.8.2. Open the warehouse rollup door by hitting the top button. Let it go all the way up. Back out the forklift and close the rollup door to prevent unauthorized entry.
- 5.8.3. Adjust the forks to maximum width to fit the drum carrier. Bring the forks up to your middle thigh. Brake on. Neutral gear. Stand between the forks, facing the right fork you will adjust first. Disengage the lock at the top of the fork. Place your left thumb at the top of the fork. Pull up the fork with your right hand just enough to relieve the pressure at the elbow of the fork. Shift your body weight to your right foot. Shove the fork out with even pressure at top, fork elbow (with left leg), and mid fork with right hand. Shove all the way out. Turn 180° to face the other fork and repeat. Remember to push fork with inside leg, or it will bind.
- 5.8.4. Drive forklift to main waste door. Raise forks so bottom is at big crossbar (about knee height) to clear bumps. Tilt full forward and lower to meet drum carrier and shove in. Tilt the forks back to retain drum carrier.
- 5.8.5. Open both bat wing arms of drum carrier. The small one on the left will stay open if you drape the locking chain back towards the forklift. The tail of the chain should dip into the chain storage cup for the light spinner chain. Roll the drum in with the drum truck. Push in as far as possible. Bring the locking chain around the front of the drum. Turn the long black latch handle counterclockwise as far as you can without dropping the pawl. Pull the chain taut and place a link between the lugs. Turn the latch handle strongly clockwise to pull in the drum and tighten the chain. The ratchet will hold. (Pull the handle tight and lift the pawl to release when done.)
- 5.8.6. Lift the forks to the crossbar again. Ensure that the top of the mast clears the bottom of the door. Check for traffic and blow horn. Back out. Drive along west edge of basketball court until you can see the street entrance of the parking lot. This ensures you are visible to approaching drivers. Raise the bottom of the forks to the big bolts on the mast to clear the dumpster.
- 5.8.7. Drive in slowly until your side of the drum is just inside the dumpster and a few inches higher.
- 5.8.8. Park the forklift and dismount to the right.
- 5.8.9. Pull the tilt-chain with both hands to swing the drum forward 180 degrees and dump the glass. Do not breathe the glass dust. Beat the bottom of the drum to

dislodge plastic bottles that may have wedged. Tilt the drum back so open side with the third chine is up. Repeat as needed.

WARNING: If the wind is blowing dust from the dumping process back towards you, use a dust filter/nuisance mask, available from the EH&S Coordinator or Hazardous Waste Specialist.

- 5.8.10. Lower the drum carrier all the way to the main waste room floor. Take the forklift back to the front of the warehouse. Close all the doors.
 - 5.8.11. Leave the last empty drum in the carrier so you can move it from a standing posture.
- 5.9. Palletizing Drums:
- 5.9.1. Place a four-way pallet, in good condition, in a parking space next to the roll up door about two inches from the curb. Place the yellow plastic ramp at the other end of the pallet. Using the curb and ramp reduces the strain required to load the drums on the pallets. Use the drum truck to move the pallets and place them so the labels face out at a 45° angle. This orientation allows the labels to be read from the front (in the waste truck) or from the side (in rows awaiting shipment). Place four drums of the same profile together to facilitate counting for the manifest and transfer to treatment facility. Next best is to group drums going to the same treatment facility. Never strap together drums whose contents are not compatible (see Corporate Safety Manual).
 - 5.9.2. Band the drums with half-inch plastic tape so they do not jump off the pallet while loading the waste truck or during shipment. Pull about seventeen feet of tape out of the banding machine and loop clockwise around the four drums on the pallet. The band should be supported by the neck of a poly drum or the chine of a steel drum. Fold ten inches of the loose end back along the length of the band. The steel buckle has two prongs that should be located points up and out. Push the loop of tape through the back of the buckle and over the prong on the same side of the buckle. Fold the running end of the tape back on itself and push it through the buckle and over the prong on its side. Position the tensioner about ten inches left of the buckle with the slots facing up. Grip the red and black handles together to lift the foot, and place the drum side band between the frame and the foot. Place the running band through both the front cutting slot and one of the middle winding slots. Lift the red handle away from the black handle to ratchet the winding slots until the band is tight. Squeeze the red and black handles again to cut the running band, and release the tensioner from the band around the drums.
 - 5.9.3. Using the forklift, stack the pallets two high in the main waste collection area, leaving aisles that extend from the roll-up door to the back of the area between

the stacked pallets.

- 5.9.4. Pallets of flammable drums such as mixed flammable solvent drums may not be stacked. They must always be on the bottom. Due to the weight, pallets of soil and pure DCM drums must always be on the bottom.
- 5.10. Shipping waste:
 - 5.10.1. Waste material must be shipped before the oldest drum is 90 days from start date. Ship every drum that is older than 14 days, so that shipments are about 75 days apart. Roughly 4 weeks before shipping, estimate the number of drums and cost of the shipment. Print the “Drum Tally” form (\\TAFS\Public\aaSacSafe\Waste 2\Drumtally) and count the number of drums for each profile. Estimate the number to be shipped by dividing current number by weeks since last shipment, and multiplying by total weeks from last shipment to next shipment (usually ten). Ship sooner depending on Main Waste storage space.
 - 5.10.2. Use the Excel spreadsheet at \\TAFS\Lab\Sacramento\Admin\aaSACSAFE\waste\counts&shipments\shipments to estimate weights and disposal costs for the shipment. Place a Purchase Order (PO) through Oracle. Use the vendor number 11439 for Clean Harbors, or 10867 for Nexeo if applicable, from the safety “Purchases” folder. The cost center for waste disposal is 320.23000, or Waste Disposal MISC. Enter items by profile name. Additional fees (state, recycling, transportation, processing) are also added. Notify the controller, lab director, and regional manager so the large purchase order may be approved as quickly as possible if not approved following day.
 - 5.10.3. Contact the waste vendor and request a waste pickup for the week preceding the 90 day date. Send your lab pack inventories and list of lab pack drums for approval and quoting. Lab packs must be approved for shipment, which requires at least two weeks. Enter the disposal codes (see Lab Pack Guidelines) on the lab pack inventories, and tops of drums. Photocopy the lab pack inventories double sided to reduce bulk in records, and place the originals on the drums. Use clear packing list envelopes so the drum number is visible.
 - 5.10.4. Send an e-mail to Sacramento - All, Sacramento-Corporate Staff and EMLab-Sacramento explaining when the waste truck will block egress by automobile from the back parking lot. On the day of shipment, install the drain mat to prevent the contents of a leaking drum from entering the storm drain. The drain is behind the parking bumper so no one will park right on top of it. The two foot square magnetic mat is kept on the personnel door in the main waste room. Place the spill cart near the drain and familiarize all participants with

the blue drum uprighting lever. Use the parking slots next to the ramp as staging area. Replace the vent bungs on the solvent drums with solid shipping bungs. Pull the flammable drums from the small shed and place on pallets. Arrange the pallets on the asphalt in a checkerboard so drums may be placed from both sides. Do a final “as shipped” count.

- 5.10.5. Have the driver back his trailer even with the cryogen fill station closest to the waste ramp. Stand where the driver can see you in the mirror and, when the trailer is within six feet close your hands together to indicate remaining distance, then place palms towards the driver to say “stop here”. Verify the driver’s identity. Give the driver a broom to sweep out any wood chips. Use the forklift to place the pallet jack in the trailer if needed. One fork should be under the handle near the bigger wheels, where the weight is. Pump the pallet jack up a bit so the forks will slide out after delivery. Lift the pallets to the lower crossbar to get them out of the building without hitting the ground. Lift to the second crossbar to fit onto trailer. When only eight feet of trailer remains, take out the pallet jack. Push the remaining pallets in place using the heavy pallet stored by the safety shower. Offer the driver five (5) DOT Placards located in the shelves to the right of the sink.
- 5.10.6. Sign and date the DOT manifest and land ban forms. Check DTSC requirements regularly. Current regulations require only one land ban form per year per profile, but you may get one per shipment. Have the driver sign. Continuation sheets are not signed. Pull your Generator copy. Make two photocopies of the front pages so that we have a completely legible copy for TestAmerica records and one to send to DTSC, PO Box 400, Sacramento, CA 95812-0400 within thirty days of shipment.
- 5.10.7. Stand at the end of the wall when the waste truck leaves, and flag down any traffic that might run into the truck. Send e-mail notification of quarantine soil shipment to USDA. Update drum logbook and waste shipment logbook. Sweep floor, and mop if needed. Vacuum spider webs from flammable solvent shed. Order steel drums, vent caps, vermiculite, yellow bags, placards, clear envelopes, drum labels, smaller drums/pails and blue drums as needed for the next cycle.
- 5.11. Soapy Wash Water: Pipes carry soapy water from laboratory sinks to the 500-gallon tank under the grate in the wash water treatment room. A pneumatic pump propels the water through a bag filter, two carbon filters, and a flow meter to plastic tank two (T-2) or three (T-3). Daily procedures are posted on the inside door of the treatment room. The logbook is kept on top of one of those two tanks. Check that pH in the tank is between 5 and 11 before discharging.
 - 5.11.1. At the beginning of each month, hold the first tank to be filled for testing; do not discharge it. Take VOA and metals samples to Sample Control. Hold the

water in that tank until testing confirms contaminants are within allowed levels. David Alltucker is the TestAmerica Sacramento Project Manager for quote 3203545. Results are kept in the file folder titled "POTW Data" in the waste coordinators desk.

- 5.11.2. Notify Joe Schairer (EH&S/Facilities) when non-conforming materials are found in the wash water sump. Note: organic materials such as latex paint, propylene glycol, or ethylene glycol may clog the carbon filters and require immediate replacement. The slug may be stored in polyethylene drums or tank one (T-1) until a determination is made.
- 5.11.3. Clean the foot valve and in-line strainer every three months. Replace the bag filter with a new one from the cabinet. Cut a slit in the dirty bag filter and remove the water with a dry/wet vacuum. Force the plastic mouth into an oval to slip off the retention disk. Cut the internal ears off the plastic mouth of the new bag. Follow the directions on the carbon drums when replacing. Attach a plastic standpipe to the outflow stub. Backfill with water overnight. Backflush one day before connecting as final drum. Use the stinger and tap water carboy to re-prime the pneumatic pump. Do not enter the sump.
- 5.12. Waste Area Inspections: The waste processor inspects the main waste storage area, the H-3 waste storage closets, the sample archive room and the waste sheds weekly using the standard inspection forms (`\\TAFS\Lab\Sacramento\Admin\aaSacsafe\Waste\Weekly Main Checklist`). Results are recorded in the safety logbook for the area.
- 5.13. Records Retention: Records are retained in accordance with the Quality Assurance Manual (QAM), maintained on the Purple Q. Several specific types of records are addressed here for convenience.
 - 5.13.1. Inspection Records: General inspection records are retained for two years. OSHA inspection records, when present, are retained for six years.
 - 5.13.2. Tracking logs, as part of the waste manifest process, are retained indefinitely.
 - 5.13.3. Test data from internal samples is retained per the records retention policy.
 - 5.13.4. Manifests and supporting/associated documentation are retained indefinitely.
 - 5.13.5. Land Disposal Restrictions, as part of the waste manifest process, are filed with and retained with the manifests.
 - 5.13.6. State or Federal reports are retained for seven years.
 - 5.13.7. Other documents. Waste profiles, as part of the manifest process, are retained

indefinitely.

5.14. TestAmerica Sacramento Waste Profile List

- 5.14.1. Flammable Solvent (Clean Harbors profile AP336072_143, collected in and shipped to in 55-gallon steel drums): Toluene, Hexane, Acetone, Methanol, Isooctane, and Methylene chloride. Note that Benzene and Diethyl ether wastes are lab packed. These drums weigh more than 350 pounds. OSHA mandated vent caps are used while in shed.
- 5.14.2. HPLC Solvents & Water (Nexeo profile 44-11304 / Clean Harbors profile AP336073_165) collected in and shipped in 55-gallon blue plastic drums: Water with acetonitrile, methanol. This waste stream is incinerated, so aqueous samples unsuitable for disposal to LLE Water due to organic contaminants may go here. Examples include “Dark liquor”. The HPLC profiles may be altered in the future to accommodate Ethylene glycol and Propylene glycol coolant solutions, if required. These drums carry a Class 9 DOT label and are incinerated.
- 5.14.3. Acid Drum (Nexeo profile 44-11305) shipped in 55-gallon blue plastic drums): Water with nitric acid, hydrochloric acid and sulfuric acid. The Nexeo profile may have RCRA levels of Chromium or Lead. The Clean Harbors profile CH94280 may also contain Arsenic. Nexeo has a \$750 shipment minimum circa 2012. These drums get Class 8 “Corrosive” DOT labels for shipment.
- 5.14.4. Soil Samples (Clean Harbors profile CH94287 collected and shipped in 55-gallon steel drums): Soil samples, ash, and sludge. This may contain USDA quarantine soils so it must be thermally treated to eliminate crop pests. USDA permit for the receiving facility must be attached to each drum.
- 5.14.5. Laboratory Trash Land Fill (Clean Harbors profile CH94284-LF collected and shipped in 55-gallon steel drums): Contaminated personal protective equipment, used filters, aluminum oxide, calcium silicate, silica gel, absorbed acid spills, and dirty glassware. These drums get Class 8 “Corrosive” DOT label and receive acid vials from the metals lab.
- 5.14.6. Laboratory Trash High VOC (Clean Harbors profile CH94284-HV collected and shipped in 55-gallon steel drums): Contaminated personal protective equipment, used filters, sodium sulfate with Methylene chloride, aluminum oxide, calcium silicate, silica gel, absorbed solvent spills, trash with Chrome 6 contamination and glassware from soil samples or those with ineradicable client identification. These drums carry a Class 9 DOT label and are incinerated.

- 5.14.7. LLE Water: This is clean, unused aqueous samples or aqueous samples that have been extracted with methylene chloride and retain about 2% DCM. Nexeo profile 44-22167 is sent to US Ecology, Beatty, Nevada, for solidification and land fill. (Clean Harbors profile CH94286 for incineration is kept active as backup)
- 5.14.8. All Lab packs: Custom pack guidelines supplied by Clean Harbors will be followed. Packed and shipped in either fiber or steel drums, as appropriate, size depends on volume of waste to be shipped). Required for samples (oil or soil), which contain over 50 ppm polychlorinated biphenyls. TestAmerica typically includes PCB standards even though our source standards are less than 50 ppm. TestAmerica also disposes the vials from advanced technology. The TCDD/TCDF spiking standards are used formulations that may be incinerated as PCB waste. Unused formulations carry "F" codes, which would preclude treatment in the United States. PCBs must be incinerated at a higher temperature and longer dwell time than other hazardous wastes to prevent dioxin formation. Hydrofluoric acid drums use "pig mat pulp" absorbent to avoid any risk of reaction. Potassium permanganate/nitric acid samples are packed in a poly drum.
- 5.14.9. Mercury Lab packs: Chemical oxygen demand (COD) waste and metallic mercury contaminated with sulfur is packed separately in plastic drums, size depends on volume of waste to be shipped, but typically are 5-gallon pails and sent to Clean Harbors.
- 5.14.10. Standard Lab packs are composed of small containers containing process wastes, samples, or excess chemicals not including mercury or PCBs. They are packed in fiber, plastic or steel drums; size depends on the volume, and shipped to Clean Harbors.
- 5.14.11. Carbon Filters (Clean Harbors profile CH94282) are 55-gallon steel drums filled with activated carbon. They are Siemens ASC-200-2 filters used to remove acetone and dichloromethane from our soapy wash water. The filter is spent when either contaminant nears 1.0 PPM or the drum bulges. Residual water is drained to the lab sink before shipment. Place the drum on the heavy pallet reserved as a pusher and rotate the standpipe counterclockwise to collect the water in a bucket or pan. Recap the 2-inch outlet stub for shipment. Each drum may last one year before replacement in June or September.
- 5.14.12. Empty steel drums (Clean Harbors profile CH101820). These are drums that were damaged during the rampacting process. State law prohibits discarding empty 55-gallon drums in the dumpster.
- 5.14.13. Medical waste (Clean Harbors profile MWCH220382). This includes

extracted blood and tissue samples, or first aid supplies contaminated with blood. These are 30 gallon or smaller fiber drums as specified by Clean Harbors.

6. RESPONSIBILITIES

- 6.1. Waste Coordinator (EH&S Manual 13.4.1). The Waste Coordinator is required to take eight-hour initial Hazardous Waste Management training, four-hour annual refreshers and an eight-hour initial Hazardous Material Management training on Department of Transportation regulations and four-hour annual refreshers. The waste coordinator performs weekly audits of the 90 day storage areas. The coordinator maintains and provides the correct in-house waste labels for drums, carboys, and all other waste containers. The coordinator reviews the compatibility of waste containers, establishes waste stream profiles, and ships hazardous, and biohazard waste. The waste coordinator prepares the following:
- Biennial Report to California Department of Toxic Substances Control,
 - Provide information to California tax preparer (Bill Nash) as required.
 - Environmental Protection Agency (EPA) generator ID as required.
 - Mail copy of manifest to California Department of Toxic Substances Control within thirty days of waste shipment.
- 6.2. Waste Processor (CSM 13.4.2): Responsibilities of the waste processor include:
- Prepare dump reports (see 5.2).
 - Pull containers from Sample Control.
 - Crush extract and VOA vials in the Vyleater,
 - Dump excess samples to the appropriate drum for shipping.
 - Use the Rampactor to crush clean glass and hazardous Laboratory Trash,
 - Pump the soapy water from laboratory sinks daily, and
 - Log, inspect, seal, label, mark and stack the drums on pallets for shipment.
- 6.3. Analyst: Labels and dates waste containers per the operational SOPs. Ensures that waste containers are emptied and the labels are updated appropriately. Follows the procedures in the operational SOPs, and as outlined here. Remove client identification from bottles. Analysts should report unusually high results to Waste Processor and Waste Coordinator (Greater than RCRA levels, greater than 50 ppm of PCB's or greater than 25 ppb TCDD/TCDF TEQ (toxic equivalent)).
- 6.4. Project Managers: E-mail the waste processor and sample administration staff within 30 days after invoicing the project if samples or extracts are to be retained longer than 30 days. PM's should enter in TALS non-standard disposal requirements.

7. REFERENCES/CROSS REFERENCES

- 7.1. Operational SOPs.
- 7.2. TestAmerica Environmental Health and Safety Manual (CW-E-H-001).
- 7.3. TestAmerica Sacramento Addendum to the EHSM (WS-PEHS-0002)
- 7.4. Sacramento Quality Assurance Manual, for current facility map (WS-QAM).
- 7.5. TestAmerica Records Retention Policy, (CW-L-P-001).
- 7.6. Manufacturer's instruction manuals for the Rampactor and Vyleater.
- 7.7. Department of Transportation security plan.
- 7.8. Clean Harbors Self-Pack Guidelines.

8. ATTACHMENTS

- 8.1. No attachments are present.

9. REVISION HISTORY

- 9.1. WS-EHS-0001 Revision 4.6, Effective 12/07/2018
 - 9.1.1. Section 3.4.1, added, "or toe guards".
 - 9.1.2. Section 5.1.1 added, "and the public drive".
 - 9.1.3. Section 5.1.3 added, "In the sample administration area, there is a 55 gallon drum for incinerable lab trash, and a 60 gallon yellow garbage can for AEEE packing materials (to be disposed as incinerable.)"
 - 9.1.4. Section 5.1.3.2 added, "Methylene Chloride waste" and "and used disposable extraction glass trash."
 - 9.1.5. Section 5.1.3.3 added, "flammable".
 - 9.1.6. Section 5.3.2.1 removed note, "There is a \$5 reward for turning in solvent drums that are properly filled."
 - 9.1.7. Section 5.4.2 revised to, "VOA vials: VOA analysts collect 40 ml vials with remaining unused sample of the purged VOA vial in cardboard boxes lined with plastic bags. Unused and expired aqueous and soil sample are removed from refrigerated storage and collected in VOA boxes. When the collection



boxes are full or after no more than one year all waste VOA vials are transferred to the main waste room. Once they are transferred to the main waste room, the processor performs the following:"

- 9.1.8. Section 5.4.3 removed, "20 L steel drum is used to collect the vials, then pour" and added "collected".
- 9.1.9. Section 5.4.4 added, "and in TALS".
- 9.1.10. Section 5.4.7 revised to, "Next to the waste label, apply a 100 mm square DOT diamond indicating hazard class: 3 and 6 for solvent, 6 for DCM, 8 for Acid, 9 for HPLC drum."
- 9.1.11. Section 5.57 revised, "under the radio" to "in the Waste Room".
- 9.1.12. Section 5.6.4 removed, "and PPQ Form 550".
- 9.1.13. Section 5.9.4 revised to, "Pallets of flammable drums such as mixed flammable solvent drums may not be stacked. They must always be on the bottom. Due to the weight, pallets of soil and pure DCM drums must always be on the bottom."
- 9.1.14. Section 5.10.1 added, "Ship sooner depending on Main Waste storage space."
- 9.1.15. Section 5.10.2 revised to, "Use the Excel spreadsheet at \\TAFS\Lab\Sacramento\Admin\aaSACSAFE/waste/counts& shipments\shipments to estimate weights and disposal costs for the shipment. Place a Purchase Order (PO) through Oracle. Use the vendor number 11439 for Clean Harbors, or 10867 for Nexeo if applicable, from the safety "Purchases" folder. The cost center for waste disposal is 320.23000, or Waste Disposal MISC. Enter items by profile name. Additional fees (state, recycling, transportation, processing) are also added. Notify the controller, lab director, and regional manager so the large purchase order may be approved as quickly as possible if not approved following day."
- 9.1.16. Section 5.11 revised to, "Soapy Wash Water: Pipes carry soapy water from laboratory sinks to the 500-gallon tank under the grate in the wash water treatment room. A pneumatic pump propels the water through a bag filter, two carbon filters, and a flow meter to plastic tank two (T-2) or three (T-3). Daily procedures are posted on the inside door of the treatment room. The logbook is kept on top of one of those two tanks. Check that pH in the tank is between 5 and 11 before discharging."
- 9.1.17. Section 5.11.1 revised to, "At the beginning of each month, hold the first tank to be filled for testing; do not discharge it. Take VOA and metals samples to

Sample Control. Hold the water in that tank until testing confirms contaminants are within allowed levels. David Alltucker is the TestAmerica Sacramento Project Manager for quote 3203545. Results are kept in the file folder titled "POTW Data" in the waste coordinators desk."

- 9.1.18. Section 5.14.2 added, "These drums carry a Class 9 DOT label and are incinerated."
- 9.1.19. Section 5.14.3 added, "These drums get Class 8 "Corrosive" DOT labels for shipment."
- 9.1.20. Removed Revision History prior to 2014, it can be found in previous versions of this SOP.
- 9.1.21. Editorial changes.
- 9.2. WS-EHS-0001 Revision 4.5, Effective 09/18/2015
 - 9.2.1. Updated copyright statement on cover page.
 - 9.2.2. Section 5.14.9 – Added chemical oxygen demand waste to sentence.
 - 9.2.3. Section 5.14.9 – Deleted sentence referencing Heritage recycling for COD waste.
 - 9.2.4. Editorial changes.
- 9.3. WS-EHS-0001, Revision 4.3, Effective 09/05/2014
 - 9.3.1. Appended to Section 3.1 – "Pipettes, broken glass and VOAs will be collected and transported in cardboard boxes to prevent lacerations. Drums will be closed and liquid removed from the top to prevent spills in transport. Secondary containment will be used in liquid transport where possible to prevent spills. Doors will be kept closed as much as possible to discourage intrusion. Lab trash buckets should be carried to the drum and the tied bag place inside with the opening facing up to minimize lacerations or spills. OSHA mandates the use of spring loaded vent bungs on solvent drums to prevent pressurization explosions."
 - 9.3.2. Appended to Section 3.2 – "The West Sacramento Fire Department may be able to provide the needed fall protection/extraction harness."
 - 9.3.3. Changed Section 3.6.2 to – "Special spill response equipment includes drum lifter lever, and UN rated 85-gallon salvage drum. Leaky drums should be placed with the top towards the top of the salvage drum to facilitate final treatment and disposal."

- 9.3.4. Replaced previous Section 5.2. with the current Section 5.2 detailing criteria for disposing of sample waste.
- 9.3.5. Changed first sentence in Section 6.1 – “Waste Coordinator (EH&S Manual 13.4.1). The Waste Coordinator is required to take eight-hour initial Hazardous Waste Management training and four-hour annual refreshers.”

	Always check on-line for validity. <h2 style="text-align: center; color: red;">Automated Storage, Retrieval, and Discarding of Samples</h2>	Level:  Work Instruction
Document number: S-SS-WI12042		
Old Reference: 1-P-QM-PRO-9015512; DOD - SOP-SS-006		
Version: 11		Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date 28-JUN-2019	Document users: 5_EUUSLA_Sample Support_Manager, 6_EUUSLA_Sample Support_ASRS	Responsible: 5_EUUSLA_Sample Support_Manager

Note: Handling of Quarantine Soils approval is through Quarantine Soil Permit P525-190620-001 - Robert Dempsey (ELLE President), permittee

[Revision log](#)
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[Purpose](#)
[Scope](#)
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[Personnel Training and Qualifications](#)
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Revision log

Revision: 11		Effective Date: <u>This version</u>	
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure B	Applicable to the procedure	Added references to the scanned locations	
Procedure G	Changes made to the process	Updated the hazardous waste stream information appearing on screen Updated the chart. Changed the Acid Waste, Hazardous & Quarantine Soil Wastes, and Soil Extracts in Sodium Bisulfate descriptions	

Revision: 10		Effective Date: <u>Mar 15, 2017</u>	
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure B.3.	Clarification	Added location of samples with Cart ID	
Procedure B.7.	Enhancement	Added the Missing Samples-Requisition Contacts email information	

Reference

Chemical Hygiene Plan, Lancaster Laboratories, current version.

Purpose

The purpose of this SOP is to define correct procedures for the storage, retrieval and discard of samples using the Automated Storage and Retrieval System (ASRS) or the manual Sample Storage program.

Scope

This SOP covers the steps required to put samples in location, pull samples for analysis, and locate samples not found in the ASRS. It also outlines the procedures used for handling samples designated as “Hold at Discard” and removing samples upon their discard date for disposal.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal and state laws and regulations.

Information concerning the known toxicity, properties, or any special handling precautions can be found in the material safety data sheets (MSDS) available in Sample Storage or by a Safety Officer. The MSDS should accompany the samples when pulled for the technical departments. Safety glasses and sample storage lab coats are required as personal protective wear.

Hearing protection devices (earplugs) are available. However, occupational noise measurements indicate that the sound pressure level is below the Occupational Safety and Health Administration’s (OSHA) Action Level of 85 dB (A). The Action Level is the threshold above which OSHA mandates hearing protection.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

The initial training consists of observing all of the steps in this procedure carried out by an experienced ASRS operator. Next, the trainee performs all of the same steps while the experienced person watches, answers questions, and gives feedback. Following the initial training, an experienced operator is available as a resource until no longer required. Operators are considered proficient when all of the steps in the procedure can be carried out independently.

Procedure

A. Daily ASRS operations/pulling requisitions:

1. Log onto the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Choose the *Pick* option in the ASRS Functions.
4. Requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled.

Choose the *Shipping* option on the Function Selection Panel and then select *Order Wave Release*, the rule that best describes the orders you wish to pull, and change the Order Status to *Batched*.

5. To release orders, select the *GO* option on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. Place the Tote ID with the order so the samples can be scanned to this ID.
6. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples to fill the order are displayed in green; scan the Sample Bar Code and the Tote ID to remove the sample from the container and link it to the designated Tote ID.

NOTE: Discards can also be done at this time. See Procedure Section E.

7. When an order is finished, missing samples are displayed on a Discrepancy List found in Parallax. Any other queued orders are also processed.

8. Completed orders are stored in the appropriate queuing area until a lab technician comes to retrieve them.

B. Locating samples not in the ASRS

1. When all of the samples are retrieved from the ASRS, check the Discrepancy List to view any samples that are still needed to complete the order.

2. Samples previously assigned to another Tote ID are located on a cart waiting to be put back into the ASRS, or have not yet been returned from the lab.
 3. Samples with a Cart ID, Bin ID, or room ID are scanned to a fixed location and can be located in the designated soil location area, in the associated laboratory fixed location, or at one of the splitting stations. Overflow carts can be found in the ASRS walk-in.
 4. If no prior destination is given, the samples are new and should be located in the SA walk-in waiting to be labeled or processed by one of the other sample support areas. If the samples were already processed by one of the other areas, the samples should be on a ASRScart waiting to be scanned to a location cart and put back into the ASRS.
- NOTE:** Samples that require a pH check, volatile prep, or homogenization must be communicated to that area prior to the samples being given to the technical areas.
5. Samples that are not located by the requisition time need to be communicated to the appropriate lab technician. Keep the technical department up-to-date on the status of missing samples. If the technician informs you that a sample is *Rush* status, the sample becomes a priority. If you require further assistance to find the sample, contact your supervisor.
 6. If the sample is not located by the end of a shift, the ASRS operator is required to let the next shift know the status of the sample. If the sample is missing on your next shift, you must contact your supervisor.
 7. If a sample is not found 24 hours prior to the deadline, the client service representative must be contacted to inform the client. These samples are indicated by the labs using the Missing Samples-Requisition Contacts email address which alerts this group that the sample must be located or a reason must be provided as to why the sample was not found.

C. Retrieving samples designated as client hold

1. Processing the Client Hold Orders
 - a. Log on to the system by typing your employee login and password.
 - b. Choose the *ASRS* option in the Function Panel
 - c. Choose the *Pick* option in the ASRS Functions
 - d. Client Hold requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled. This is done by choosing the *Shipping* option on the Function Selection Panel. Then select *Order Wave Release*, chose the *Client Hold* orders, and change the status of the orders you wish to pull to *Released*.
- NOTE:** For Client Hold orders the *CLH* option must be selected as the conveyance type.
- e. When orders are released, the *GO* option is selected on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. The Tote ID is placed with the order and the samples are scanned to this

ID. If multiple orders for the same client are placed in the same tote all Tote IDs must be displayed.

f. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples needed to fill the order are displayed in green. Scanning the Sample Bar Code and the Tote ID removes the sample from the container and links it to the designated Tote ID. If a container is filled before the order is complete you can request a new Tote ID by scanning the next sample, using the mouse and right clicking on the proper order on the Put Bar and select *New Tote ID*.

NOTE: Discards can also be done at this time. See Section E.

g. When the order is finished, the box containing the samples must be taken over to the bulk workstation. There the samples are imported into the Client Hold area by inducting the Tote into an available location.

2. Scanning Totes in to the Client Hold Pod

a. Using the RF gun log in to the system.

b. On the Main Menu choose the *Ship* option.

c. Next choose *Matrix Induct*.

d. Scan the Tote you wish to put away and scan an Empty shelf location in the Client Hold Pod. You will get a question to Consolidate “Yes or No”. Always answer “Yes”.

e. If multiple Tote IDs are on the current box, scan each ID to the same location. The last ID scanned contains all the samples in the box. All previous IDs scanned must be crossed off with a sharpie so that only the last ID scanned is displayed.

NOTE: If a container is scanned to a location that is not full and has the same client and account number as a Tote you are currently scanning away, you can combine by:

f. Repeating steps 1 – 3 above.

g. Scan the Tote you wish to combine to the location in which the container resides.

h. All the samples from the existing container are transferred to the container ID that was recently scanned to the location; all samples need to be moved to the current box.

3. Discarding from containers in the Client Hold Pod

a. Remove the container from the designated location and write the shelf location on the container ID (this is to allow for the container to be returned to the proper location if all the samples aren't up for discard at this time).

b. Using the Evolution workstation, choose the *Bulk* option on the Function Selection Panel.

- c. Navigate to Execute and Deplenish Container.
- d. Choose the Existing Container option.
- e. Scan the container ID.
- f. Scan each sample located in the container. There are three scenarios that could take place:
 - (1) Sample is obsolete - remove from the container as normal and discard.
 - (2) Sample is obsolete and hazardous - remove from container and place on a designated hazardous cart.
 - (3) Sample has not reached the necessary time to be marked as obsolete and remains a client hold. In this case the system prompts you to scan a container. The options are:
 - (a) Scan the existing container. All samples designated as client hold remain in the current container.
 - (b) Scan an existing container that is scanned to a different Client Hold Location. All samples designated as Client Hold move to this container (samples must be same client and account number).
 - (c) With either option the container scanned remains unless a new container to discard from is selected.
- g. Return the container to the designated shelf location if samples remain. If no samples remain the container ID must be removed and the box is not returned to the Client Hold Pod.

D. Sample put away

1. Log on to the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Select the *Put Away* option in the ASRS Functions.
4. Choose *Put Away*, which displays the available empty locations for each cell configuration listed in order of container height.
5. Choose the container size you need and type the number of samples and press ENTER. This brings you the available containers for the size requested.
6. When the containers arrive, scan the container ID. The grid display on screen shows the available open locations.
7. Scan the Sample ID and the Open Location ID to code the sample away.

NOTE: Procedure D. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

E. Sample discard procedure

NOTE: Discards can be processed through Parallax. In the SA function, choose *Download ASRS Discard File*. This starts a count of the samples that are up for discard and sends the file to *Evolution*.

1. Log on to the system by typing your employee login and password.
2. Choose the *ASRS* option in the Function Panel.
3. Select the *Deplenish* option in the ASRS Functions.
4. Select the *Discard* option which displays the available discards for each cell configuration listed in order of container height.
5. Choose the container size you wish to discard from and type the number of samples you need and press ENTER. This brings you the available containers for the size requested.
6. When the containers arrive, scan the container ID and the grid displays on the screen. The regular obsolete samples are displayed with blue lines going through the sample; hazardous are in red.
7. Scan the designated samples and separate upon removal from the ASRS and take to the appropriate area for disposal.

NOTE: Procedure E. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

F. Disposing non-hazardous samples

1. Use discretion when disposing all non-hazardous samples. Do NOT discard a solid or liquid sample if it has an “off odor” or unusual appearance.
2. Liquid samples should essentially be clear and odorless like water. It is acceptable for the sample to have some discoloration and/or sediment present. Unless the sample has a solvent odor, an oily or viscous appearance, a paint odor or appearance, a lot of solids, etc., it may be poured into the discard sink. The ventilation hood and the cold water tap must be turned on. After pouring the sample out, place the container in the trash hopper.
3. Discard solid, non-hazardous samples into the trash hopper. Do not dump solid samples out of their containers. Discard containers unopened, with the contents intact, directly into the hopper.
4. If you have any doubt on the proper disposal of a sample, place it on the cart designated for questionable discard and notify your supervisor or the EHS (Environmental Health and Safety) Group for assistance.

G. Disposing of hazardous samples

1. Take the hazardous discard cart to the Sample Storage workstation.

2. Log on to Parallax.
3. Using the SA function on the tool bar, choose the *Sample Storage* option.
4. When in this program, choose *Hazardous Discard Assessment*.
5. Scan the barcode on one of the hazardous samples.
6. If the sample discard date has not come up yet, a window appears indicating "Sample has not been discarded." If applicable, the hazardous results are displayed. Before disposing, further investigation must be done to see if the sample can be discarded.
7. If the sample does **not** have any Final Results that exceed the adjusted Hazardous Limit for that particular analysis, a window appears indicating "**No hazardous analysis in this sample.**" Read the lab notes very carefully at this point because the sample may be hazardous for reasons other than the analyses performed.
8. If the sample has no hazardous analyses or lab note indicating hazard, check the matrix of the sample and the sample designation to be sure that the sample matrix is not something that must not be dumped down the sink (i.e., oils, paint, or paint thinner, ink, etc.).
9. If the sample is flagged as *Hazardous* and the discard date has passed, the following information appears on the screen:

Master Analysis Number
 Piece Analysis Number
 Analysis Name
 Final Result
 Reporting Units
 Discard Date
 Adjusted Hazardous Limit

Waste Stream

10. Based on the data provided from the Assessment Program, the sample needs to be put into one of the appropriate waste streams which are indicated in the program and have fixed locations assigned in the hazardous assessment area.

Waste Stream	Description
Acid Wastes	pH <4; no heavy metals; dumped in acid neutralizer

Waste Stream	Description
Solvent Wastes	A liquid flagged as hazardous due to an analysis result regulated under TCLP Waste Characteristic List (except heavy metals); dumped in solvent waste drum
Labpack Wastes	A solid or liquid flagged as hazardous that does not fit into one of the other categories
Hazardous & Quarantine Soil Wastes	A soil from USDA regulated counties(soil)
Waters containing Heavy Metals (free liquid)	Liquid flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver); liquid poured off into a drum
Waters containing Heavy Metals (in vials)	Liquid samples in vials flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver)
Waters containing Cyanides	Sample vials flagged for cyanide-containing compounds
PCB-containing Soils	Solid samples (and containers) flagged for PCBs
PCB-containing Water & Oils	Liquid samples (and containers) flagged for PCBs
Soil Extracts in Methanol	Place vials in open-head drum labeled: "GC/HPLC and 40-mL vials containing organic solvents"
Soil Extracts in Sodium Bisulfate	Place vials in open-head drum labeled: "Corrosive Organic Extracts"

11. If you have any doubt on the proper waste stream for disposal of a sample, place it on the cart designated for questionable hazardous discard and notify your supervisor or the EHS (Environmental Health and Safety) Group for assistance.

12. Lancaster Laboratories reserves the right to return samples to the submitting client if they contain high levels of hazardous substances or do not fit into one of our existing waste streams.

End of document

Version history

Version	Approval	Revision information
9	22.APR.2011	
10	01.MAR.2017	
11	28.JUN.2019	

Appendix H

Data Package Review Checklist

Location –

Jobname -

Lab –

Lab Report No. -

Note – the following checklist is intended to allow an organized review of the draft laboratory data package for compliance with the laboratory SOP and general data usability. Draft results and the supporting raw data will not be deleted or discarded. Comments herein will be provided to the laboratory who will generate a final laboratory data package and an electronic disk deliverable (EDD) (if needed). The EDD will be uploaded to the EIM database and the Data Review Module (DVM), an automated data review program, will proceed. The DVM program checks QC measures (e.g. RPR) and applies standard letter qualifiers to the results for a sample or batch of samples, for failure to meet QC criteria. Additional letter qualifiers may be applied to the data as outlined in the following discussion.

NARRATIVE – confirm target compounds and note if QC criteria not being met

RESULTS SUMMARY –

Confirm sample results (approximately 20%) reported in data package match against the edd

Compare results between Form I in data package against certificate in data package.

Confirm at least 1MS/MSD per batch of samples analyzed (provided sample volume provided)

Method Blanks – less than 1/3 the reporting limit – further evaluated using DVM. If sample concentration < 5X method blank, sample concentration will be qualified as B (not present substantially above the level found in the method blank)

Field Reagent Blanks – to be evaluated using DVM program – sample concentrations < 5X field blank concentrations will be qualified as B (not present substantially above the level found in field blanks)

Location –

Jobname -

Lab –

Lab Report No. -

LCS concentrations must be rotated from high to mid from batch to batch
Mid- and High-LCS (recovery should be 70-130%)
DVM may qualify result if recovery is outside limits

Low-LCS (recovery should be 50-150%)
DVM may qualify result if recovery is outside limits

Surrogate Standard (recovery should be 70-130%)
DVM may qualify sample result if outside limits and sample not diluted by more than 4 times.

Internal Standard

The IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration and must not deviated by more than 70-140% from the most recent CCV standard. Usability limits are 50-150% for both ICAL and CCV standards. Manually qualify sample result if outside usability limits.

FIELD DUPLICATES – Criteria is 20% when both results are $> 5X$ the RL. When one or both results are below $5X$ the RL, compare the difference between the results to the RL. When criteria are exceeded, the sample and field duplicate results will be qualified J and considered to be estimated.

MATRIX SPIKE/MATRX SPIKE DUPLICATE RECOVERY – spike recovery must meet criteria (within lab limits) unless sample concentration is $> 4X$ spike concentration. Spike value alternates from batch to batch.

DVM may qualify sample result if outside criteria.

COC REVIEW/SAMPLE RECEIPT

Samples relinquished by field

Samples received at lab next day

Samples packed in wet ice

Sample temperature upon receipt (not frozen to 10 C)

Location –

Jobname -

Lab –

Lab Report No. -

HOLD TIME – Hold time is 14 days from date of collection to extraction; 28 days from extraction to analysis. Data will be evaluated if samples analyzed outside of hold time. Professional judgement will be used to determine if results should be qualified as estimated (J/UJ).

Check for:

Initial calibration (minimum 5 point for linear; 6 points for quadratic)
 The origin must be forced through zero (specified by method 537/537.1).
 Reporting Limit CCV (recovery should be within 50%)
 May qualify sample result if CCV is outside criteria

Check Standard (ICV) – Second Source; conc should be at mid-level of the curve (70-130%)
 May qualify sample result if ICV and bracketing CCVs are outside criteria

CCV (recovery should be within 30%)
 May qualify sample result if sample between CCVs is outside criteria

LC/MS/MS OPERATING CONDITIONS

The target analyte ions should be within 0.3 m/z of the expected mass.
 PFOA Scanning method – 413-369 transition
 415-370 (Internal Standard)
 HFPO-DA scanning method 285.00 > 169.00
 13C-HFPO-DA 287.00 > 169.00
 See method for scanning methods of other compounds

INITIAL CALIBRATION

Standard Concentration	Criteria for Native Analyte	Criteria for Surrogate	Criteria for IS
Lowest calibration standard ≤ MRL	± 50% of true value	± 30% of true value	20% RPD
> MRL	± 30% of true value	± 30% of true value	20% RPD

Ion ratio $\leq 50\%$
 Qualify positive results if ion ratio is greater than 50%

COMMENTS

Reviewer -

Date -

Appendix I

Electronic Data Deliverable (EDD) Format

Chemours/DuPont Standard EIM EDD Format

Revisions to DuPont Standard EIM EDD Format Dated 11/7/13

Inclusion of Chemours. Standard EIM EDD Format description applicable to both Chemours and DuPont

Introduction

The Chemours/DuPont Corporate Remediation Group (CRG) maintains a corporate environmental database that stores field data, analytical results, QA/QC results, water levels, and other information resulting from the activities of Chemours/DuPont environmental projects. Much of this data is provided by analytical labs or sampling contractors performing analytical and sampling services for Chemours/DuPont. Chemours/DuPont has implemented the Locus Environmental Information Management (EIM) system as the corporate database. To optimize loading data generated by these contractors, an EDD file format has been developed for importing laboratory analytical data into the Locus EIM database. Following is a description of the Locus EIM EDD specification (EIM EDD) for Chemours/DuPont contractors.

General EDD Information

In general, EIM EDDs will be uploaded by the laboratory that does the sample analysis. Locus EIM user accounts and training will be provided to laboratories. The EIM EDD must match the hardcopy report in terms of samples, tests, analytes, and results. Also, Chemours/DuPont generally requires the lab composite results such that only one result is reported for each analyte (i.e., the lab submits only the result judged best when a sample is re-analyzed for particular analytes due to exceeding calibration range, etc.). However, there may be cases where regulations require results from all runs be submitted. These cases will be specified by the project chemist during project setup.

Normally, all data for a particular sample delivery group will be contained in one file. This group is normally referred to as a lot (or group), which makes up a normal reporting/invoicing group and usually consists of samples for a given project and site that the lab has received in one day, including all associated QC samples and results. Note that QC results may be contained in more than one EDD if field samples from different lots were analyzed in the same QC batch.

Samples taken for matrix spike/matrix spike duplicates (MS/MSD) and laboratory replicates (REP) are QC samples that have field samples, and are subject to the following controls:

1. If the field sample is from Chemours/DuPont and is in the current lot for the current project, then:
 - a. The parent or un-spiked sample and result information should be included in the EIM EDD and;
 - b. The FIELD_SAMPLE_ID for the MS, MSD, and REP samples should be included for those records. If the Chemours/DuPont sample is used as the parent for the MS/MSD/REP, the field sample ID must be the same as parent sample ID. There should be no MS, MSD, or REP in the FIELD_SAMPLE_ID. For example, for an MS sample, the FIELD_SAMPLE_ID must be NR0513-LHWABLDG (same as parent) not NR0513-LHWABLDG-MS.
2. If the parent field sample is not from a Chemours/DuPont site, or is from a Chemours/DuPont site but not the current site and project, then:
 - a. The field sample and result should not be included, and;

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- b. The FIELD_SAMPLE_ID must be null for the MS, MSD, and REP samples, but these QC samples must have the ORIGINAL_LAB_RESULT result as per the spec.
3. Lab originated (QA/QC) samples such as lab control spikes or method blanks should not have a FIELD_SAMPLE_ID populated in the edd.

QA/QC results involving relative percent recoveries and relative percent differences, e.g. MS/MSDs, REPs, lab control spikes and lab control spike duplicates (LCS/LCSD), and surrogates must also include these recoveries and differences plus the maximum and minimum recoveries and differences that are acceptable, as applicable. For example, an MS sample requires a result, the relative percent recovery, and the maximum and minimum permissible relative percent recovery. An MSD sample requires a result, the relative percent recovery, the relative percent difference, the maximum and minimum permissible relative percent recovery, and the maximum permissible relative percent difference.

EDD Specification Details

The following list outlines the requirements associated with generating EDDs for Chemours/DuPont's implementation on Locus' EIM system.

- The EDD must be an ASCII file with no header or footer.
- Each record must be alike with respect to format.
- Every analytical result is represented by a single record.
- The record format of the EDD is positional and therefore, each field must be listed in the order specified in Table 1.
- The length of each field must not exceed the width specified in the "Length" column of Table 1, or the data will be truncated.
- Every field must be separated by a semi-colon.
- Null or blank fields must be delimited.
- Each record (last record excluded) must be terminated with a carriage return.
- Required fields are indicated in **bold** in Table 1.
- Non-required fields may be populated depending on the project circumstances, or the particular data being reported. These requirements are described in Table 1 in the "Field Contents" column and in footnotes at the bottom of table.
- The column titled "VVL" represents if a data field contains lookup valid values. These values are provided in the valid value attachment and can be accessed in EIM through your Lab View.
- No data in any field in the EDD should be enclosed in quotation marks.

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Table 1: The EDD record format is defined as follows:

Table 1. Chemours/DuPont EIM EDD format				
Field	Field Name	Length	VVL	Field Contents
1	SITE_ID	10	Yes	Identification ID assigned to the project site in EIM. Has list of values. This will be supplied by the project chemist.
2	FIELD_SAMPLE_ID	C30	No	Field Sample number or identifier. Must be left blank for lab-originated samples (e.g., lab control samples, method blanks, blank spikes, etc.). Should be populated for lab duplicates and matrix spikes and duplicates (if the sample that is spiked is the client sample).
3	LAB_ID	C10	Yes	Code or identifier for a lab. Has list of values. This will be supplied by the project chemist.
4	ANALYTICAL_METHOD	C30	Yes	Analytical method used. Has list of values.
5	ANALYSIS_DATE	Date	No	Date of analysis, MM/DD/YYYY.
6	ANALYSIS_TIME	Time	No	Time of analysis (HH:MM), military time.
7	PARAMETER_CODE	C12	Yes	Analyte CAS Number or other code (for those parameters that do not have a CAS Number). Has list of values. For TICS (RESULT_TYPE_CODE = TIC): <ul style="list-style-type: none"> • If a positive identification is not made (e.g., Unknown), use "TIC" for PARAMETER_CODE and report PARAMETER_NAME, concentration and retention time as appropriate. • If a positive identification for a TIC is made, use the CAS Number of the identified constituent and report PARAMETER_NAME, concentration and retention time as appropriate. • If no TICs are found, use "NOTICS" for the PARAMETER_CODE, "No TICs Found" as PARAMETER_NAME, "NA" for RETENTION_TIME (Field 17) and "ND" for LAB_RESULT (Field 9). • If reporting a Targeted TIC, use EVS number (CASNO created by Chemours/DuPont) for PARAMETER_CODE. Report parameter name as compound (targeted TIC). Example ALLYL ALCOHOL (Targeted TIC). If compound not detected enter "NA" in RETENTION_TIME (Field 17) and "ND" in LAB_RESULT (Field 9). If compound detected, report concentration and retention time as appropriate. RESULT_TYPE_CODE should be set to "TRG".
8	RESULT_TYPE_CODE	C5	Yes	Code identifying the type of result (TIC, SU, SPK, etc.). Has list of values.
9	LAB_RESULT	C10	No	Analytical result. Required of all samples except surrogates and spikes. If not detected, enter the laboratory reporting limit here (MDL or PQL as appropriate). If detected above the MDL and below the reporting limit, enter the result in this field and a "J" in LAB_QUALIFIER. Laboratory will only report one result per sample per parameter unless otherwise instructed by client. Refer to description for Field 7, PARAMETER_CODE for reporting results for TICs.
10	DETECT_FLAG	C1	Yes	Coded value (Y or N) indicating whether an analyte was detected in the sample. Required all analytical results.
11	LAB_UNITS	C10	Yes	Unit of measure of the result. Has list of values. Enter the units associated with the entry in the LAB_RESULT or SPIKED_RESULT column.
12	METHOD_DETECTION_LIMIT	C10	No	Method detection limit. For PQL projects and TICs, leave null. Required for all non-spiked samples for MDL projects.

Table 1. Chemours/DuPont EIM EDD format				
Field	Field Name	Length	VVL	Field Contents
13	LAB_REPORTING_LIMIT [LAB_DETECTION_LIMIT] – Column Name in EIM	C10	No	Actual reporting limit (i.e., PQL) realized by the lab, adjusted for preparation, dilution, etc. Required for all non-spiked samples. For TICs leave NULL.
14	LAB_MATRIX	C10	Yes	Matrix of sample as analyzed by the lab. Has list of values.
15	LAB_SAMPLE_ID	C20	No	Internal ID assigned by lab to a sample.
16	LAB_QUALIFIER	C10	No	Laboratory qualifier. Qualifier must match lab report. If a laboratory qualifier is entered in the EDD, this qualifier must also appear in the laboratory report, and visa versa.
17	RETENTION_TIME	Time	No	Retention time (MM:SS), required for TICS only. For others enter NA or leave blank.
18	DILUTION_FACTOR	C7	No	Dilution factor if the sample was diluted.
19	PREP_METHOD	C20	No	Preparation method (if applicable).
20	PREP_DATE	Date	No	Date of preparation MM/DD/YYYY (if applicable).
21	PREP_TIME	Time	No	Time of preparation HH:MM (if applicable).
22	ANALYSIS_LOT_ID	C20	No	Laboratory analysis batch number or ID.
23	INSTRUMENT	C20	No	Lab defined identifier for instrument on which analysis was performed.
24	PREP_AMOUNT [INITIAL_PREP_AMOUNT] – Column Name in EIM	C10	No	Amount of sample used in the preparation.
25	PREP_UNITS [INITIAL_PREP_AMOUNT_UNITS] Column Name in EIM	C10	Yes	Unit or measure of sample preparation amount. Has list of values (Lab_Unit valid values).
26	PREP_AMT_BASIS	C5	No	The basis of the weight of the amount of the sample prepared: W or D are the only valid values.
27	SAMPLE_DELIVERY_GROUP	C20	No	Laboratory sample delivery group (i.e., lot).
28	LAB_BLANK_SAMPLE_ID	C20	No	ID of laboratory method blank that is associated with the sample identified in the FIELD_SAMPLE_ID and/or LAB_SAMPLE_ID fields. Can be left blank if only one method blank is run with a given prep or analysis lot.
29	ERROR	C10	No	+/- 2-sigma error (pertains to radiological results only)
30	PARAMETER_NAME	C60	No	Name of parameter. Any correct synonym is acceptable. TICs may have values such as Unknown, Long Branch Alkane, etc. If no TICs found, report "No TIC Found".
31	ANALYSIS_TYPE_CODE	C5	Yes	Coded value specifying type of analysis (e.g., Initial, Reanalysis, Re-extraction, Dilution, etc.). Has list of values. INIT is most common type.
32	FILTERED_FLAG	C1	Yes	Flag to identify whether sample was filtered in the field or by the lab. The only valid values are Y or N.
33	LEACHED_FLAG	C1	Yes	Flag to identify whether sample was leached prior to being analyzed. The only valid values are Y and N.
34	LEACHATE_METHOD	C20	Yes	Method used to leach a sample (if applicable).
35	LEACHATE_DATE	Date	No	Sample leachate date MM/DD/YYYY (if applicable).
36	LEACHATE_TIME	Time	No	Sample leachate time (if applicable) HH:MM, military time.
37	SAMPLE_PREP_LOT_ID	C20	No	Laboratory prep lot number or ID (if applicable).
38	LEACHATE_LOT_ID	C20	No	Laboratory leachate lot number or ID (if applicable)

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Table 1. Chemours/DuPont EIM EDD format				
Field	Field Name	Length	VVL	Field Contents
39	<i>SAMPLE_DATE</i>	Date	No	Date sample was collected (field sample) or created in the lab (lab generated QC samples): MM/DD/YYYY
40	SAMPLE_PURPOSE	C5	Yes	Coded value identifying purpose of the sample. (e.g., regular sample, Lab Control Samples, Lab Control Sample Duplicates, Method Blanks, Lab Duplicates or Replicates, etc.) or lab-transformed samples (e.g., Matrix Spikes and Duplicates). Has list of values.
41	<i>ORIGINAL_LAB_RESULT</i>	C10	No	The concentration of the analyte in the original (unspiked) sample. Should be populated only for matrix spikes and duplicates (MS, MSD, and REPs).
42	<i>SPIKE_ADDED</i>	C10	No	Amount of spike added to sample. Applicable only to spiked samples or surrogates.
43	<i>SPIKED_RESULT</i>	C10	No	Concentration of the analyte in the spiked sample. Applicable only to spiked samples or surrogates.
44	<i>SPIKE_RECOVERY</i>	C10	No	Percent recovery. Applicable only to spiked samples or surrogates.
45	<i>RPD</i>	C10	No	Calculation of relative percent difference (applicable only to matrix spike duplicates, lab control sample duplicates, and lab replicates or duplicates).
46	<i>RPD_LIMIT</i>	C10	No	Upper limit for RPD (percent) (applicable only to matrix spike duplicates, lab control sample duplicates, and lab replicates or duplicates).
47	<i>UPPER_LIMIT</i>	C10	No	Upper spike recovery control limit (in percent). Applicable to surrogates or spiked samples only.
48	<i>LOWER_LIMIT</i>	C10	No	Lower spike recovery control limit (in percent). Applicable to surrogates or spiked samples only.
49	<i>LAB_ARRIVAL_DATE</i> [LAB_RECEIPT_DATE] - Column Name in EIM	Date	No	Date that the sample arrived at the lab (mm/dd/yyyy). Required for field samples only.
50	<i>LAB_ARRIVAL_TIME</i> [LAB_RECEIPT_TIME] – Column Name in EIM	Time	No	Time that the sample arrived at the lab (HH:MM). Required for field samples only.
51	<i>HARD_COPY_DUE_DATE</i>	Date	No	Hardcopy lab report due date.
52	<i>RUSH_TAT</i>	C1	No	Specify if sample was submitted as “Rush” – valid values for this field are Y and N.
53	<i>EDD_DUE_DATE</i>	Date	No	Date (mm/dd/yyyy) the EDD (electronic data deliverable) is due.
54	<i>SUBCONTRACT</i> [ANALYSIS_SUBCONTRACTED_FLAG] – Column Name in EIM	C1	No	Enter Y (Yes) if analysis was performed by a subcontractor lab. Otherwise, field can be left blank. The only valid values are Y or NULL.
55	<i>SUBCONTRACT_LAB_ID</i>	C10	Yes	Code or identifier for the subcontract lab. Has list of values. Prior approval is required by client to use subcontract lab. Client will provide Subcontract_Lab_ID.
56	LAB_REPORTING_LIMIT_TYPE	C10	Yes	Coded value identifying the type of reporting limit (e.g., practical quantitation limit, method detection limit, etc.). Only valid values are PQL or MDL.
57	BASIS	C3	Yes	Basis for reporting the result. Only valid values are W, D, or N.

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Table 1. Chemours/DuPont EIM EDD format				
Field	Field Name	Length	VVL	Field Contents
Notes: a. Fields in Regular font are required for all records (e.g., <i>LAB_ID</i>). b. Fields in <i>Italic font</i> are required for various subsets of samples and/or analyses. c. Fields In Regular font are optional.				

Appendix J

Data Validation Standard Operating Procedure

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate Polyfluorinated Alkyl Substance (PFAS) data generated by “Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)” (EPA Method 537.1, Version 1.0, November 2018). Validation will be performed to assess compliance of the sample data to the reference method and any applicable Work Plans, Quality Assurance Project Plans (QAPPs), and/or SOPs. In addition, the usability of the PFAS data provided by the analytical laboratory will be determined based on the general guidance provided in the “National Functional Guidelines for High Resolution Superfund Methods Data Review (USEPA, April 2016) and “Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537” (EPA 910-R-18-001, November 2018).

The validation findings will be presented in a quality assurance review (QAR) or data usability summary report (DUSR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I’s), including any changes to the analytical results and data qualifier codes, or a data summary spreadsheet of the qualified analytical results, will be included in the support documentation of the QAR.

2.0 EVALUATION TOOLS

- Field duplicate form (DVF_DUP_537.xlsm)

Chemistry Applications:

- Curve fitting software (DVF_CAL.xlsm) and Gretl

3.0 REFERENCE DOCUMENTS

- “National Functional Guidelines for High Resolution Superfund Methods Data Review (USEPA, April 2016).
- “Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537” (EPA 910-R-18-001, November 2018).
- US EPA Method 537.1, Version 1.0 (November 2018).
- Guidance for Labelling Externally Validated Laboratory Analytical Data for Superfund Use (EPA 540-R-08-005, 2009).

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4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the PFAS data based on an evaluation of information presented in the data package deliverables. Compliance to the aforementioned method (and Work Plan, QAPP, and/or SOPs, when applicable) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the compliance assessment will be described in terms of comments/deficiencies about the data/deliverables and presented in two subdivisions (*i.e.*, Reporting Issues and Procedural Issues) of the Organic Data Evaluation Section of the QAR. Each issue discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer may contact the project laboratory to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by the client). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the Reporting Issue subdivision of the QAR and included as an attachment to the QAR.

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4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the PFAS data based on an evaluation of the information presented in the data package deliverables. The findings of the PFAS data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in the data tables to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination (B), unusable results (R/UR), estimated results (J/UJ), tentative identifications of target compound results (N), and a general qualifier for all results reported below the limit of quantitation (if applicable).

The data reviewer's criteria for evaluating the usability of the PFAS data and the resultant qualifications will be as stipulated on the attached Table for the Validation of PFAS Data Generated. Additional qualifications will be assigned based on professional judgement. It should be noted that the project manager should be consulted when "professional judgement" use is indicated on the attached table.

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	≤ 10 °C, but not frozen, by Method and/or other criteria as listed in Work Plan or other project document.	<p>If the samples were received frozen/broken use professional judgment to determine how to qualify data.</p> <p>If temperature is > 10 °C (or project criteria) but ≤ 20 °C (or 2× project criteria), qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If temperature is > 20°C (or 2× project criteria), qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p> <p>Note time of collection relative to receipt at laboratory. Professional judgement should be used if < 8 hours has elapsed from collection to receipt at the laboratory and evidence of cooling is present to determine if qualification due to elevated temperature applies.</p>
Sample Preservation	The preservation reagent Trizma® (5.0 g/L), is added to each sample bottle as a solid prior to shipment to the field or prior to sample collection as a buffering reagent and to remove free chlorine.	If a chlorinated water sample was not preserved with Trizma, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).
Technical Holding Time	<p>Aqueous samples should be extracted within 14 days of collection.</p> <p>All extracts should be analyzed within 28 days after extraction.</p>	<p>If a holding time is exceeded, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If a holding time is grossly exceeded (<i>i.e.</i>, > twice the holding time), qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p>

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Initial Calibration	<p>Internal standard (IS) calibration technique should be used to generate a first or second order calibration curve forced through zero with at least five standard concentrations. When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70 - 130% of its true value. The lowest calibration point should calculate to be within 50 - 150% of its true value.</p> <p>The peak asymmetry factor should be calculated and within 0.8 - 1.5 for the first two eluting chromatographic peaks in a mid-level calibration standard every time a calibration curve is generated.</p>	<p>When evaluating initial calibration, use professional judgment to first assess impact of any out-of-criteria labeled PFAS on the corresponding target PFAS(s).</p> <p>If the low point or consecutive points at the low end of the curve are below the lower recovery criteria, qualify positive results < lowest compliant point as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If the low point or consecutive points at the low end of the curve are above the upper recovery criteria, qualify positive results < lowest compliant point as estimated (“J”).</p> <p>If a point in the middle of the curve or non-consecutive points (besides the low point) quantitate outside of criteria, qualify positive results as estimated (“J”).</p> <p>If the high point or consecutive points at the upper end of the curve quantitates outside of criteria, qualify positive results > highest compliant point as estimated (“J”).</p> <p>Professional judgement should be used to qualify “not-detected” results as estimated (“UJ”) if low recoveries are observed for standards other than the lowest calibration point.</p> <p>Professional judgement should be used to qualify positive results as estimated (“J”) or “not-detected” results as estimated (“UJ”) if the peak asymmetry factor criteria is not met.</p>

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Quality Control Sample (QCS) (Second Source)	Laboratory to analyze at least quarterly or when preparing new standards, as well as during the Initial Demonstration of Capability (IDC). The QCS may or may not be provided with each data set due to the required frequency. Results must be within 70 - 130% of true value.	<p>Qualification is for all samples associated with initial calibration standards being verified.</p> <p>When evaluating QCS, use professional judgment to first assess impact of any out-of-criteria labeled PFAS on the corresponding target PFAS.</p> <p>If target PFAS has %R > 130%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If target PFAS has %R < 70% but ≥ 10%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If target PFAS has %R < 10%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p>

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Continuing Calibration Check (CCC) (See Note #1 for additional information.)	<p>Initial calibration is verified by analyzing a low level (at the MRL or below) CCC prior to analyzing samples. CCCs are then injected after every 10 samples and after the last sample, rotating concentrations to cover the calibrated range of the instrument.</p> <p>Recovery for each analyte and surrogate must be within 70 - 130% of the true value for all but the lowest level of calibration. Recovery for each analyte in the lowest level CCC must be within 50 - 150% of the true value and the surrogate must be within 70 - 130% of the true value.</p> <p>The absolute areas of the isotopically labeled IS should be within $\pm 50 - 150\%$ of the average areas measured during the initial calibration.</p>	<p>Qualification is for all samples on both sides of the out-of-criteria calibration verification standards.</p> <p>When evaluating calibration verification, use professional judgment to first assess impact of any out-of-criteria labeled PFAS on the corresponding target PFAS.</p> <p>If target PFAS has %R > 130% (or > 150% for lowest level CCC), qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If target PFAS has %R < 70% (or < 50% for lowest level CCC) but $\geq 10\%$, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If target PFAS has %R < 10%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p>

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Blanks (See Note #2 for additional information.)	Summarize all results greater than the method detection limit (MDL) or minimum reporting level (MRL) present in the blanks (depending on whether reporting to MDL or MRL). The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target PFAS is found in the blank but not in the associated sample(s), no action is required. If a target PFAS is found in the blank and a sample result is between the MDL and the MRL, qualify the positive result as “not-detected” (“B”) and raise the MDL to the value of the original result. If a sample result is $\leq 5\times$ the blank result, qualify the positive result as “not-detected” (“B”) and raise the MDL and/or MRL (if lower than original reported positive result) to the value of the original result. If a sample result is $> 5\times$ the blank result, qualification is not required.
Surrogate Standards (Labelled Analytes, spiked prior to extraction)	Surrogate standards may consist of $^{13}\text{C}_2$ -PFHxA, $^{13}\text{C}_3$ -HFPO-DA, $^{13}\text{C}_2$ -PFDA, and d_5 -NEtFOSAA and are added to all calibration standards, QC, and samples. Recoveries must be 70 - 130% of the true value.	Qualification will be applied to all target PFAS for any out-of-criteria surrogate recovery unless the data reviewer determines that the qualification should be limited to a subset of target PFAS based on professional judgement. If the recovery is $> 130\%$, qualify positive results for all associated PFAS as estimated (“J”) and do not qualify “not-detected” results. If the recovery is $< 70\%$ but $\geq 10\%$, qualify positive results for all associated PFAS as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If the recovery is $< 10\%$, qualify positive results for all associated PFAS as estimated (“J”) and qualify “not-detected” results as unusable (“R”). Professional judgement should be used to qualify results in more dilute analyses of the same sample extract if surrogate recoveries are outside of limits in the least dilute analysis. In addition, professional judgment should be used if the IS used to quantitate the surrogate recovery is outside of criteria.

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Internal Standards (Labelled Analytes, spiked prior to analysis)	Peak area counts for all IS in all injections must be within $\pm 50\%$ of the average peak area calculated during the initial calibration and 70 - 140% from the most recent CCC.	If an IS response is outside of 50 - 150% of the average initial calibration response, but $\geq 10\%$, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ") for all PFAS quantitated using that IS. If an IS response is outside of 70 - 140% of the most recent CCC response, but $\geq 10\%$, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ") for all PFAS quantitated using that IS. If an IS response is $< 10\%$ of the average initial calibration response or $< 10\%$ of the most recent CCC response, qualify positive results for the associated PFAS as estimated ("J") and qualify "not-detected" results for the associated PFAS as either estimated ("UJ") or unusable ("R") based on professional judgment (evaluating the approximate signal-to-noise ratio for the IS and the expected response for the target PFAS at the LOQ).
Laboratory Fortified Blank (LFB)	The laboratory is to analyze at least one LFB daily or one for each extraction batch of up to 20 field samples and rotate between low, medium, and high concentrations. Results of LFB analyses must be 70-130% of the true value for each method analyte for all fortified concentrations, except the low concentration ($\leq 2 \times$ MRL). Results of the LFBs corresponding to the low concentration for each method analyte must be 50-150% of the true value.	The LFB qualification will be applied to all associated samples (all samples analyzed that day or all samples in the same extraction batch, whichever is more frequent). Data reviewer must determine whether the spiked concentration is applicable to the samples being evaluated (e.g., if a low-level LFB is spiked $<$ reported MRL, it may not be able to be used for data quality evaluation). If the recovery is $> 130\%$ (or $> 150\%$ for low-level), qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is $< 70\%$ (or $< 50\%$ for low-level), qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If the recovery is $< 10\%$, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as unusable ("R").

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Table for the Validation of PFAS Data

Quality Control Item	Usability Criteria	Action
Laboratory Fortified Sample Matrix (LFSM) and Laboratory Fortified Sample Matrix Duplicate (LFSMD; if performed)	The laboratory is to analyze one LFSM and one FD or LFSMD per extraction batch (20 samples or less) fortified with method analytes at a concentration close to but greater than the native concentration, if known. A LFSMD may be substituted for a FD when the frequency of detects are low. Recoveries at mid and high levels should be within 70 - 130% and within 50 - 150% at the low-level fortified amount (near the MRL). Method analyte RPDs for the LFSMD should be $\leq 30\%$ at mid and high levels of fortification and $\leq 50\%$ near the MRL.	<p>The LFSM/LFSMD qualification will only be applied to the unspiked parent sample, and if a field replicate of the native sample was collected and analyzed, any data qualification based on the MS/MSD results shall also be applied to the field replicate.</p> <p>Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of a PFAS is $> 4\times$ the spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision.</p> <p>If the recovery is $> 130\%$ (or $> 150\%$ for lowest fortification level), qualify positive results in the native sample as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If the recovery is $< 70\%$ (or $< 50\%$ for lowest fortification level) but $\geq 10\%$, qualify positive results in the native sample as estimated (“J”) and qualify “not-detected” results in all associated samples as estimated (“UJ”).</p> <p>If the recovery is $< 10\%$, qualify positive results in the native sample as estimated (“J”) and qualify “not-detected” results in the sample as unusable (“R”).</p> <p>If the precision exceeds the RPD criterion, qualify positive results in the native sample as estimated (“J”) and do not qualify “not-detected” results.</p>
Field/Laboratory Duplicate (See Note #3 for additional information)	Project-specified criteria (RPD $\leq 30\%$ when PFAS are detected in one sample \geq sample-specific MRL). The RPD should be $\leq 30\%$ for results $> 5\times$ the sample-specific MRL. The difference between results should be $\leq 1.5\times$ MRL when at least one result is $\leq 5\times$ MRL.	<p>If both results are $<$ MRL, a quantitative assessment of duplicate precision is not performed.</p> <p>Use the MDL (or MRL if MRL-reporting was used) as a numerical value for any “not-detected” result in the difference calculation.</p> <p>If the criteria are not met, qualify positive results for the out-of-criteria PFAS in the original sample and its duplicate as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p>

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Quality Control Item	Usability Criteria	Action
Compound Quantitation and Qualitative Identification (See Note #4 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution or re-extracted with a lower volume.	<p>If a target PFAS result exceeds the instrument calibration range, qualify positive results as estimated (“J”).</p> <p>Use professional judgment to determine whether sample reanalyses and dilutions should be compared to the original analyses. If criteria (see field duplicate usability) between the original sample results and the reanalysis sample results are not met, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If a target PFAS is <LOQ but ≥ MDL, qualify positive results as estimated (“J”).</p> <p>Use professional judgment to determine whether quantitation and qualitative identifications are accurate and whether data qualification (estimated, “J”; tentative, “N”, and/or unusable, “R”) is necessary. For example, for target PFAS where both branched and linear isomers are being quantitated, professional judgement should be used to evaluate whether positive results should be flagged estimated (“J”) because the composition of the sample is significantly different than the composition of the calibration standard.</p> <p>Secondary ion transitions and ratios should also be evaluated if provided by the laboratory, although not required by Method 537.1. Positive results associated with out-of-criteria ratios (as defined by the laboratory) should be qualified as estimated (“J”) using professional judgement.</p>
System Performance	Professional judgement should be used when assessing the degradation of system performance during analyses.	Use professional judgment to qualify the data if it is determined that system performance degraded during sample analyses.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	<p>Use professional judgment to determine the need to qualify data not qualified based on the QC previously discussed.</p> <p>Write a brief narrative to give the data user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.</p>

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1. If instrument instability (*i.e.*, several calibration verification standards with PFAS exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential calibration verification standards, “not-detected” results may be qualified as estimated (“UJ”) due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (*i.e.*, an increase in instrument sensitivity).
2. The frequency of equipment/rinse blanks is determined during the sampling event. The results of an equipment/rinse blank should be applied to all samples collected in the same day by the same techniques, unless only one blank was collected for a several-day sampling event. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant.

Method blank contamination should be applied to samples in the preparation batch.

Instrument blank contamination should be applied to samples bracketing the contaminated instrument blank.

Blanks should also be evaluated using professional judgment for non-target interference.

3. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision;

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therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Laboratory duplicate results and field duplicate results apply only to the original sample and the laboratory/field duplicate.

4. When comparing sample re-analyses and dilutions to the original analyses, if a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the MRL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

Poor chromatographic performance may affect qualitative identification and/or quantitation. Indications of substandard performance include:

- High background levels
- Extraneous peaks
- Loss of resolution
- Peak tailing or peak splitting that may result in inaccurate quantitation

The laboratory analyzes a qualitative standard that contains linear and branch chained isomers of PFOA for analyses. The analysis of this standard is used to demonstrate where the branch chained isomers elute and is not included in the calibration curve. This will assist the chemist in the integration of branched isomers of PFOA in samples. Professional judgement should be used to qualify sample results as estimated due to the presence of significant branched isomer concentrations relative to the initial calibration standards (linear only) used for PFOA quantitation.